

# ORGANIC FIBER FILTERS

PART I: A METHOD OF PRODUCING ORGANIC FIBER FILTERS  
FOR AEROSOL SAMPLING

PART II: A SURVEY OF SOURCES OF ORGANIC FILTER MATERIAL  
APPLICABLE TO ATMOSPHERIC SAMPLING

*Prepared for:*

GEOPHYSICS RESEARCH DIRECTORATE AIR FORCE CAMBRIDGE RESEARCH CENTER  
AIR RESEARCH AND DEVELOPMENT COMMAND UNITED STATES AIR FORCE  
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**ORGANIC FIBER FILTERS**

**Part I: A Method of Producing Organic Fiber Filters for Aerosol Sampling**

**Part II: A Survey of Sources of Organic Filter Material  
Applicable to Atmospheric Sampling**

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**GEOPHYSICS RESEARCH DIRECTORATE  
AIR FORCE CAMBRIDGE RESEARCH CENTER  
AIR RESEARCH AND DEVELOPMENT COMMAND  
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## Final Report

### ORGANIC FIBER FILTERS

Stanford Research Institute, under Contract No. AF 19(604)-2644 with Air Force Cambridge Research Center, was given the problem of finding or developing an organic fiber filter which could be used to collect samples of air-borne particulate material from the stratosphere and upper troposphere. The collected material was to be studied by neutron activation and other radiochemical methods, so the filters were to be capable of being ashed and were to be of high purity with respect to metallic contaminants. They were to collect, with essentially 100% efficiency, solid particles in the size range 0.01 to 1  $\mu$  in diameter at a face velocity of 14 cm/sec and at a pressure drop not exceeding 110 mm of water. Also, they were to measure 24 in. by 70 in. and were to be strong enough to be handled by untrained personnel. The fiber material was to weigh between 3.5 and 4.5 g/sq ft.

Development of a technique for preparing and testing such filters is given in Part I of this report, together with a detailed description of the construction and operation of pilot plant facilities. A survey of sources of organic filter material is presented in Part II.

#### Part I

##### A METHOD OF PRODUCING ORGANIC FIBER FILTERS FOR AEROSOL SAMPLING

###### A. Introduction

A literature survey and visits to various laboratories and fiber manufacturers revealed that although fiber filters had been prepared which would probably meet the specifications, with the possible exception of purity requirements, no such filters were readily available in the United States. A technique for preparing such materials was therefore developed and studied in these laboratories, and, at the request of Air Force Cambridge Research Center, 3,000 sq ft of the material was prepared and shipped to them.



## B. Summary

A technique was developed for producing organic fibers by dispersing solutions of organic polymers into air. When a jet of a sufficiently viscous solution of a polymeric material was introduced into a high-velocity air stream, the jet was broken up and drawn out into fibers from which the solvent evaporated. The dried fibers could be collected to form a mat which served as a highly efficient filter either with or without compression, but the filters were weak and required reinforcement. A variety of polymers and solvents was studied. There was a tendency in some cases for beads to form along the fibers. In other cases, the fibers were too tacky, too weak, or too brittle. The solution of polystyrene in methylene chloride produced fibers which seemed to be particularly promising for use as filters, and this system was studied in detail.

Filter efficiencies were determined for solid aerosol particles in the size range 0.01 to 1  $\mu$  in diameter. Minima were observed in the particle size-collection efficiency curves at about 0.1  $\mu$ . Compression of the mat somewhat increased collection efficiency and greatly increased the pressure drop across the mat. An increase in the median fiber diameter was obtained either by increasing the polymer concentration or by decreasing the pressure of the air stream.

The following method was used to prepare the 3,000 sq ft of filter material in 24- by 70-in. sections: A solution containing 10 g of polystyrene per ml of methylene chloride was sprayed into air by means of a fiber generator developed for the project, using a positive pressure of 50 psi of air. The fibers were collected by suction on a Dacron net slightly larger than 24 by 70 in., and 3 generators were needed to form an evenly distributed mat of this size. When the required amount of fiber had been collected, another Dacron net was placed over the face of the filter, forming a sandwich. The sandwich was compressed with a glass roller and was cut to size. Fibers with a median diameter of about 0.4  $\mu$  were produced, and the collection efficiency of the material was about 99% for particles in the 0.01- to 1- $\mu$  diameter range at a face velocity

of 14 cm/sec at atmospheric pressure. Efficiencies of samples of filter pads produced by this method were tested for collection of particles 0.088 and 0.026  $\mu$  in diameter, at General Mills Laboratories. Atmospheric pressure, and a pressure of about 0.3 atmosphere, were tested over a range of face velocities. The material was reported to have a 100% collection efficiency for both particle sizes at atmospheric pressure, and at face velocities ranging from about 10 to 240 cm/sec. The collection efficiency for both particle sizes was also reported to be 100% at 0.3 atmosphere, and at face velocities ranging from about 25 to 500 cm/sec. In addition to these tests, calculations indicated that filter collection efficiencies would be higher at lower-than-atmospheric pressure.

The Dacron net was used to reinforce the filter and to promote ease of handling, and could easily be removed before analysis without a significant loss of fiber material. Filtered air was used in the fiber-forming process, and care was taken to keep the filters as free from contamination as possible. Metallic contaminants in the fibers ranged from about 0.1 to 10 ppm by weight, with only calcium and silicon in the upper range. The filters were crated in lots of 20, and were shipped to Air Force Cambridge Research Center.

This method of producing fibers is not new except possibly for the present purpose, and various patents have been issued which involve this technique as part of one or more claims. A description of methods, including spray methods used by other organizations to form organic filter material, and a discussion of patents relating to spray methods, is given in Part II of the report.

### C. Development of Process

#### 1. Preliminary Tests of Methods of Fiber Production

Preliminary experiments were made on 2 methods of preparing superfine fibers\* from polymers which were soluble in some organic solvent.

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\*The term "superfine fibers" is used in this report to indicate fibers which range up to 1  $\mu$  in diameter.

One of these methods consisted of coating a pair of flat surfaces with a viscous solution of the polymer, pressing the surfaces together and then pulling them apart to draw out a mass of small fibers. It was possible to produce fibers with a mean diameter somewhat below  $1\ \mu$  by this method, although with a wide range of sizes. Extensive studies would have been required to develop a filter pad by this method, and it was replaced by a method based on the fact that fibers may be formed if a sufficiently viscous solution of polymeric material is introduced into a high velocity gas stream. A generator was constructed for producing fibers by this technique. It consisted essentially of a central jet through which the polymer solution was fed, and a concentric orifice for a high velocity air stream. Filaments of polymer solution were produced by shear at the gas-liquid interface. The solvent subsequently evaporated, yielding a suspension of fibers which were carried along by the air stream. The fibers could then be collected by directing the air stream through a suitable screen. The generator used in these preliminary tests utilized an 0.018-in.-ID liquid jet and an 0.063-in.-diameter orifice for the gas stream. The polymer solution was fed to the jet under a positive pressure of 20 to 50 psi. The gas pressure was controllable from 50 to 1000 psi.

A variety of different combinations of polymer, solvent, and plasticizer were tested for superfine fiber production with this generator, using various polymer concentrations and gas-to-liquid ratios. The polymers utilized were cellulose acetate, cellulose nitrate, polymethyl methacrylate, polystyrene, polyvinyl acetate, polyvinyl chloride, and polyvinyl formal. The solvents included acetone, methyl ethyl ketone, chloroform, ethyl acetate, and methylene chloride-ethanol and ethyl ether-ethanol mixtures. Polymers and solvents were obtained from commercial sources and were used without further purification.

Superfine fibers were produced most readily from solutions of polystyrene and polymethyl methacrylate (Lucite). Mean fiber diameters of  $0.5\ \mu$  were obtained, with a range of 0.2 to  $1\ \mu$ . Under some conditions, beads as well as filaments were formed. In the tests of polystyrene and Lucite, the highest ratio of filaments to beads was obtained with the most

concentrated and viscous solutions. However, concentrated solutions yielded larger mean fiber diameters than dilute solutions. On this basis a high degree of polymerization would be desirable to give a high viscosity for a given concentration. However, the elastic properties of the polymer solution and the variation of viscosity with rate of shear would also be important, and the optimum degree of polymerization would not necessarily be the maximum available.

A solution consisting of 15 g of Lucite moulding powder in 100 ml of methylene chloride produced fibers which were of uniform diameter, but about 30% of the filter material was in the form of droplets. A solution of 15 g of Lucite moulding powder in 100 ml of its monomer produced fibers of a highly variable size range up to 1  $\mu$  in diameter, but again about 30% of the material was in the form of droplets. The most satisfactory Lucite fibers were obtained from 15 g of the Lucite moulding powder, 50 ml of its monomer, and 50 ml of methylene chloride. These fibers appeared to have a uniform diameter below 0.5  $\mu$  (about the limit of resolution of the microscope), and only about 10% of the material was in droplet form. However, the fibers were very brittle and fragile.

A solution consisting of 10 g of polystyrene per 100 ml of methylene chloride produced fibers over a wide range of diameters below 1  $\mu$ . Essentially no droplets were formed, the material was very flexible, and it was considerably stronger than the Lucite fiber material. The use of polystyrene to produce the organic fiber filters appeared very promising, and was studied more extensively in subsequent work. These studies led to the use of this material in the formation of the 3,000 sq ft of filter called for in the contract.

The gas feed to the fiber generator was dry nitrogen at a pressure of 300 psi in most of these preliminary tests, and the polymer solution was fed to the jet under a positive pressure of 50 psi. Additional tests showed that there was little difference in performance if the gas feed to the generator and the solution were both under 50 psi pressure. A new generator was designed on this basis. This new apparatus was used

for all subsequent tests, and for the preparation of the 3,000 sq ft of polystyrene filter material.

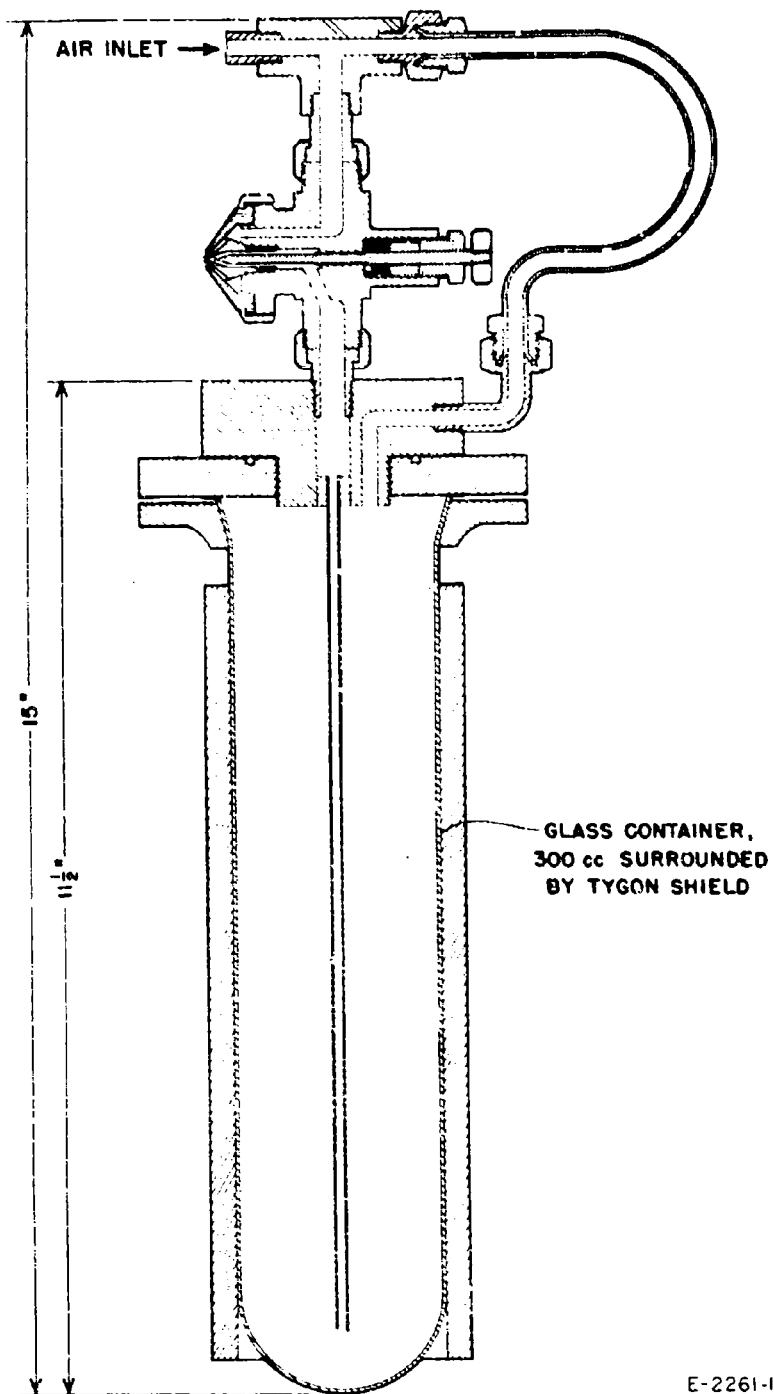
## 2. Studies of Polystyrene Fiber Filters

### a. Formation of Test Filter Pads

The apparatus used for preparing polystyrene fibers is shown in Figure 1. The same type of nozzle was used as had been used in the preliminary experiments. It was made of stainless steel and was manufactured by the Spray Engineering Company of Burlington, Massachusetts.\* The solution of polymer in an organic solvent was placed in the container below the nozzle. This container was "teed" into the air line to the nozzle so that both the solution and the entrance to the nozzle air-jet were subjected to the same pressure (usually 50 psi). Thus the solution was forced through the nozzle orifice into the air, where it was disrupted by the annular blast of air. A Tygon tube, of 2-1/2 in. O.D. with a 3/16-in. wall, was mounted around the glass container to protect the operator in case the container should shatter under high pressure.

In order to restrict the stray fibers and evaporated solvent to a small volume, the fibers were prepared in a 10-cu in. metal chamber which is described in detail later in the report. The fibers produced were drawn by suction against Dacron netting supported in a vertical position on a wire screen about 4 ft in front of the generator. Spent air was vented to the open atmosphere. Fiber pads made by this method were very weak, and were very low in pressure drop across the pad and in efficiency for collecting particles in the 0.01- to 1- $\mu$ -diameter range; the mats were generally pressed with the Dacron mesh in place by rolling them between a glass plate and a roller filled with shot in order to increase strength and improve the surface properties. Tests of the effect of compression are given later in the report. A second Dacron net was often placed on the opposite side of the mat from the first to increase the ease of handling and storage of the pads.

\*Model 125, adjustable nozzle, 1-in.-diameter cone, F2081.



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FIG. 1  
FIBER GENERATING APPARATUS

b. Methods of Testing Filter Efficiency

The efficiencies of the filters for collecting air-borne particles were determined, using lithium fluoride and ammonium sulfate aerosols. The lithium fluoride aerosols were prepared by dispersing aqueous solutions with an aerosol generator of the nebulizer type (1). The newly formed aerosols were passed through a bed of glass beads to remove the larger particles, and subsequent evaporation of the water from the droplets produced a suspension of solid lithium fluoride particles. Lithium fluoride was used because it is relatively nonhygroscopic. Several size ranges of lithium fluoride particles were employed. The largest particles were obtained by dispersing a saturated solution, and the size distribution of the particles produced in this manner, as determined from electron micrographs, is shown in Figure 2. Smaller particles were obtained from more dilute solutions, and the number median size, as would be expected, was found experimentally to be proportional to the cube root of the concentration.

The ammonium sulfate aerosols were prepared as follows. Monodisperse sulfuric acid aerosols were prepared by controlled condensation of sulfuric acid vapors. The size of the droplets produced under a given set of conditions was determined by allowing the particles in a sample to "grow" by absorption of water vapor at a controlled humidity, followed by a size determination with a light-scattering technique (2). Aerosols having droplets of the desired size were then allowed to react with ammonia, using the technique described by Robbins and Cadle (3).

Collection efficiencies were determined by measuring particle number concentrations in a stream of aerosol before and after it passed through the filter, at the same time measuring the flow rate and the pressure drop across the filter. Number concentrations of particles were determined with a General Electric Nuclei Counter (4,5). Efficiencies were expressed as 100 (final concentration/original concentration).

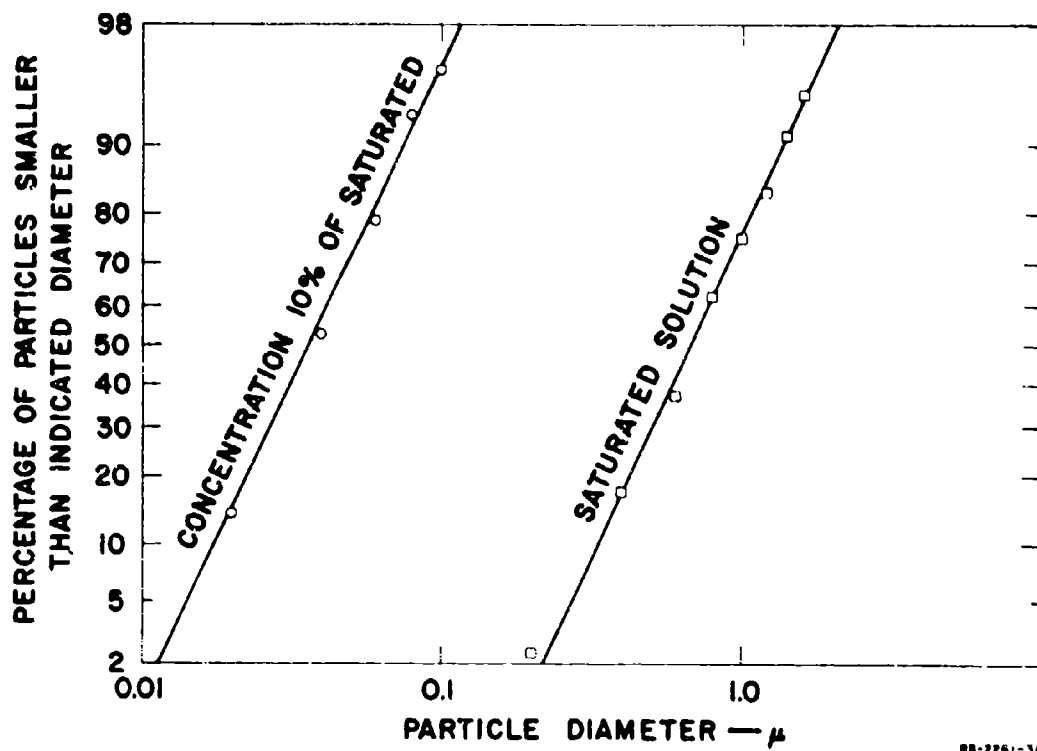


FIG. 2  
SIZE DISTRIBUTION OF LITHIUM FLUORIDE AEROSOLS PREPARED  
FROM AQUEOUS LITHIUM FLUORIDE SOLUTIONS



c. Method of Testing Filter Tearing Resistance

Internal tearing resistance was measured by the ASTM standard method D-689, using an Elmendorf Tearing Tester (Cat. No. 60-100, Thwing-Albert Instrument Company, Penn Street at Pulaski Ave., Philadelphia, Pennsylvania).

d. Methods of Photographing Fibers and Fiber Formation

The distribution of fiber sizes in test filter pads was obtained from electron micrographs, using an RCA type EMC electron microscope. Short-exposure-time photomicrographs of the jet leaving the nozzle were obtained with a Beckman and Whitley framing camera equipped with a 24-in.-focal-length objective. The camera was focussed on the virtual image of the jet, the image being produced by a 158-mm, f/6.3 "microtessar" lens. Axial, parallel, back illumination was produced by the discharge of a 5- $\mu$ f capacitor, charged to 5 kv, passed through 0.0019-in. Nichrome wire at the focal point of a 4-in.-diameter simple lens. The exposure time was about  $1/3$   $\mu$ sec per frame.

e. Experimental

Electron micrographs of some of the fibers, prepared from a 6% solution of polystyrene in methylene chloride, are shown in Figure 3. Fiber diameter distributions were obtained from micrographs, and the effect of polymer concentration and generator pressure on the distributions are shown in Figure 4. As would be expected, increasing the polymer concentration and decreasing the pressure increased the median diameter. The distributions shown in Figure 4 did not include bundles of fibers. When these were included, the median diameters were increased to 0.6, 0.9, and 3.0  $\mu$  for the 6, 10, and 20% solutions at 50 psi, respectively, and to 0.9  $\mu$  for the 10% solution at 20 psi.

A photograph of air-borne fibers being drawn to the collecting screen is given in Figure 5. The fibers were not produced by the generator as continuous filaments, but as segments varying from 2 or 3 mm to 3 or 4 cm in length.

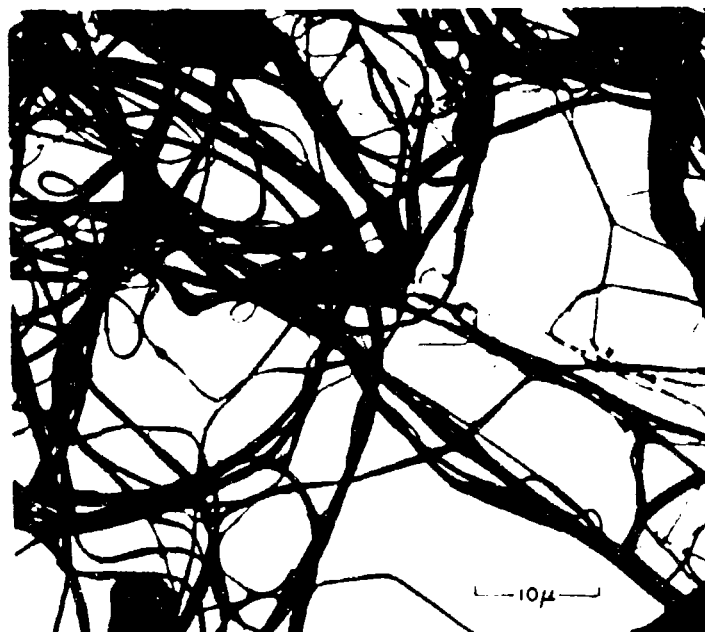


FIG. 3  
ELECTRON MICROGRAPH OF FIBERS PRODUCED AT 50 LBS PRESSURE  
FROM A 6 PERCENT SOLUTION OF POLYSTYRENE IN METHYLENE CHLORIDE

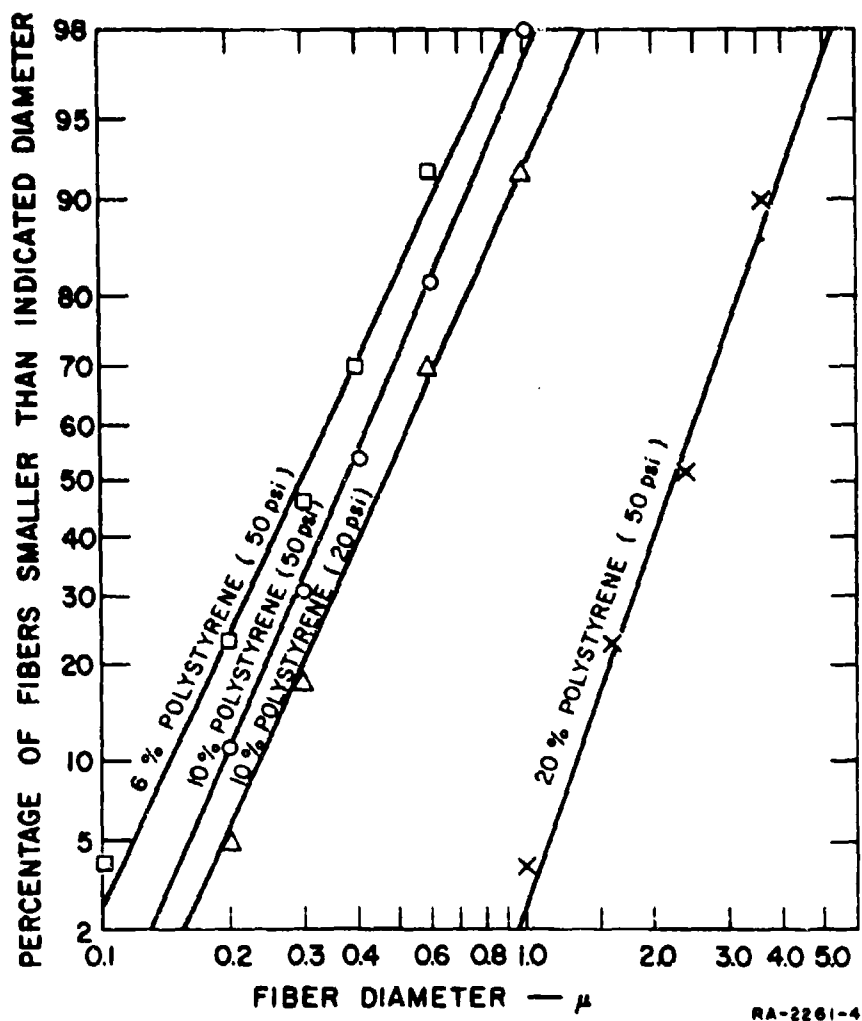


FIG. 4  
DIAMETER DISTRIBUTIONS OF FIBERS OBTAINED FROM SOLUTIONS OF POLYSTYRENE  
IN METHYLENE CHLORIDE AT VARIOUS CONCENTRATIONS AND AIR PRESSURES

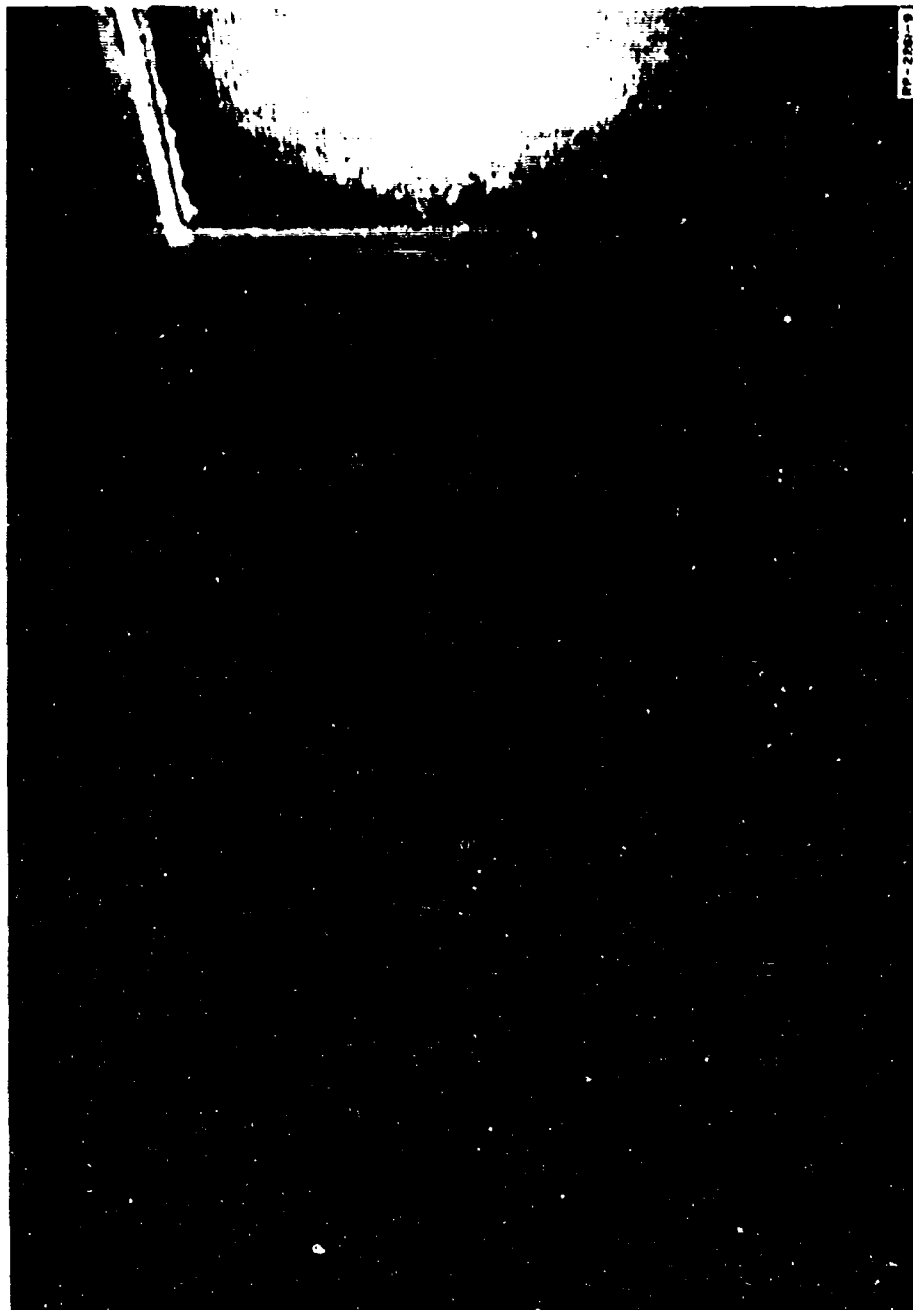


FIG. 5  
NEWLY-FORMED AIR-BORNE FIBERS BEING DRAWN TO COLLECTING SCREEN

The efficiencies of several filters at atmospheric pressure are shown in Tables I and II. The generator pressure was 50 psi, and the flow rate through the filter was 14 cm/sec in all cases. The filters measured 2 ft by 2 ft, and compression was accomplished by rolling the pads 4 times between a glass plate and a 35-lb roller filled with shot. The roller was 40 in. long and 2 in. in diameter. The results in Table I show that for particles in the range 0.01 to 0.06  $\mu$  diameter the efficiencies always exceeded 90%. Compressing the polystyrene fiber mat somewhat increased the collection efficiency and greatly increased the pressure drop. German fiber filters made by a similar process and Chemical Warfare Service Filter paper #6 are included for comparison. The results in Table II show that there was a particle size of about 0.1  $\mu$  which resulted in lowest efficiency ("maximum penetration"). The value for the lowest efficiency increased with decreasing fiber size, that is, with decreasing concentration of polymer in the solvent, and with increasing pad thickness.

Additional efficiency tests of Stanford Research Institute filters were made at the Mechanical Division of General Mills, Inc., as part of a concurrent study of filter efficiency for Air Force Cambridge Research Center (6). A solution of 10 g of polystyrene per 100 ml of methylene chloride was used in preparing the filters. The filters were rolled 4 times. The aerosol collection efficiency was determined as a function of face velocity at atmospheric pressure and at about 0.3 atmosphere. The particles were 0.088 and 0.026  $\mu$  in diameter. The filters were reported to have 100% collection efficiency for both particle sizes at atmospheric pressure, and at face velocities ranging from about 10 to 240 cm/sec. The collection efficiency for both particle sizes was also reported to be 100% at 0.3 atmosphere, and at face velocities ranging from about 25 to 500 cm/sec.

All of the filters without the Dacron support were very weak. A filter prepared from a solution of 10 g of polystyrene in 100 ml of methylene chloride and having a weight of 0.006 g/sqcm had a tearing resistance of 8 g. This is at least an order of magnitude smaller than the tearing resistance of conventional chemical laboratory filter papers. For example, the tearing resistance of Whatman #40 paper is about 100 g.

Table I

FILTER EFFICIENCIES  
Atmospheric Pressure

Filter Material	Conc. in Solvent (g/100 ml Solvent)	$\Delta P$ , <sup>a</sup> (mm H <sub>2</sub> O)	Relative Humidity (%)	Aerosol Type	Median Particle Diameter	Efficiency (%)	F (mm <sup>-1</sup> )
Polymethyl methacrylate, backing present, no compression	20	11	50	LiF	0.04	99.6	50
	20	11	50	LiF	0.01	99.9	63
	20	11	100	LiF	0.01	99.4	46
	25	9	50	LiF	0.04	97.5	41
Polystyrene, backing removed, no compression	6 <sup>b</sup>	33	50	LiF	0.04	99.1	14 <sup>c</sup>
	6	29	50	LiF	0.04	98.0	13
	8	34	50	LiF	0.04	98.8	13
	10	22	50	LiF	0.04	98.4	19
	12	22	50	LiF	0.04	95.0	14
	12	10	50	LiF	0.04	92.9	27
	20	8	50	LiF	0.04	99.5	67
	6	125	50	LiF	0.04	99.8	5.0
Polystyrene, backing removed, compressed	6	125	0	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0.06	99.9	6.2
	6	120	50	LiF	0.04	99.8	5.2
	8	120	50	LiF	0.01	99.2	4.0
	8	120	0	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0.06	99.7	4.8
	12	100	50	LiF	0.04	99.6	5.5

Table I (Continued)

Filter Material	Conc. in Solvent (g/100 ml Solvent)	$\Delta p$ , <sup>a</sup> (mm H <sub>2</sub> O)	Relative Humidity (%)	Aerosol Type	Median Particle Diameter	Efficiency (%)	F (mm <sup>-1</sup> )
German fiber d filters	Not Known	14	50	LiF	0.04	95.5	20
#1		33	50	LiF	0.04	95.5	9.0
#2		52	50	LiF	0.04	99.8	12
#3		52	50	LiF	0.01	98.2	7.5
#3		52	0	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0.06	97.0	6.6
#4		82	50	LiF	0.04	99.3	7.0
#4		82	0	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0.06	100	-
CWS #6 Asbestos	-	110	0	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0.06	99.96	7.1
	-	110	50	LiF	0.01	99.0	4.2

<sup>a</sup> Generator pressure, 50 psi. Pressure drop across filter measured at 14 cm/sec linear flow.

<sup>b</sup> Solvent was 50% monomer and 50% CH<sub>2</sub>Cl<sub>2</sub> by volume.

<sup>c</sup> Solvent was CH<sub>2</sub>Cl<sub>2</sub>.

<sup>d</sup> Delbag-Luft filter, Berlin.

Table II

## COLLECTION EFFICIENCY AS A FUNCTION OF FIBER AND PARTICLE SIZE

Atmospheric Pressure

Median fiber diameter ( $\mu$ )	Filter density (g/sq cm)	P (mm H <sub>2</sub> O)	Efficiency (%)			F (mm <sup>-1</sup> )		
			0.01 $\mu$ <sup>a, b</sup>	0.1 $\mu$ <sup>c</sup>	1.0 $\mu$ <sup>c</sup>	0.01 $\mu$	0.1 $\mu$	1.0 $\mu$
2.0	0.0022	10	68	8	90	11	0.83	23
2.0	.0105	17	97.2	57	99.0	21	5.0	27
0.4	.0027	120	97.2	85.7	100	3.0	1.6	--
0.4	.0050	408	99.9	98.3	100	1.7	1.0	--
0.3	.0032	680	99.2	99.2	100	0.71	0.71	--

<sup>a</sup>LiF aerosols<sup>b</sup>Particle diameter<sup>c</sup>(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> aerosols

As mentioned previously, filters to be processed for Air Force Cambridge Research Center were to be as free as possible from metallic contamination. Principal sources of contamination would be (1) impurities in the polystyrene and methylene chloride, (2) impurities in the air forced through the generator, (3) impurities arising from abrasion of metal surfaces by the air forced through the generator, (4) impurities in the air which was drawn by suction from outside the chamber through the wire screen on which the filter mat was fabricated, and (5) impurities picked up from interior surfaces of the chamber by the circulating air. No effort was made to prepare clean filter pads in early tests of filter fabrication, and the pads were all dirty. This was not surprising, as the pads served as rather efficient filters for the air drawn through the chambers during the fabrication process. However, methods of producing filters as free from contamination as reasonable were developed when the process was sufficiently well established to consider pilot plant operation for preparing the 3,000 sq ft of filter. It was found that the bulk polystyrene and methylene chloride had very low concentrations of metallic



contaminants, and did not require further purification. A column containing glass wool and two "Fulflo" filters connected in series with a "Sprayit" filter and pressure regulator in the line from the air compressor were sufficient to remove measurable amounts of impurities from air forced through the generator. Two Cambridge "Absolute" filters connected in parallel removed measurable amounts of impurities in air which was drawn by suction from outside the chamber. In addition, special precautions were taken to maintain cleanliness inside the chamber and in the area for processing the mats into filters. A comparison of the analysis of (1) bulk polystyrene, (2) a filter pad prepared when unfiltered chamber air was used, and (3) a filter pad prepared when filtered chamber air was used, is given in Table III. Analysis was by emission spectrography. Filter pads prepared with special precautions to maintain cleanliness did not contain a significantly greater amount of impurities than the bulk polystyrene did, and it was concluded that there was no measurable contamination arising from the air or from abrasion of surfaces in the generator. Filter pads prepared with unfiltered air contained contaminants which were generally at least an order of magnitude higher in concentration than was obtained with filtered air. Control analyses were conducted during the pilot plant operation, and are discussed later in the report.

#### D. Fiber Formation and Filtration Mechanisms

Aerosol formation by airblast atomization involves at least three stages. In the first stage, small disturbances are initiated on the surface of the jet of liquid. In the second, the aerodynamic forces acting on the disturbances draw out numerous fine fibers. In the third, the fibers collapse into droplets as a result of surface tension. Presumably, fibers rather than particles result from the present operation because the surface tension cannot overcome the viscous forces resulting from the polymer in the solvent. The above mechanism is substantiated by the complex appearance of many of the fiber images in the electron micrographs, and by photomicrographs of the liquid jet leaving the nozzle.

Table III

EMISSION SPECTROGRAPHIC ANALYSIS OF (1) BULK POLYSTYRENE,  
 (2) FILTER PAD PREPARED WITH UNFILTERED CHAMBER AIR,  
 (3) FILTER PAD PREPARED WITH FILTERED CHAMBER AIR

(ppm by weight)<sup>a</sup>

	Bulk Polystyrene <sup>b</sup>	Polystyrene Pad Prepared with Unfiltered Chamber Air <sup>c</sup>	Polystyrene Pad Prepared with Filtered Chamber Air <sup>c</sup>
Al	0.6	25	2
Ba	-	-	-
Ca	-	30	10
Ci	-	0.8	0.04
Cu	0.3	0.6	0.1
Fe	0.6	12	1.0
Mg	0.3	32	0.7
Mn	-	0.7	0.07
Na	-	-	-
Ni	-	-	-
Pb	-	6	-
Si	6	70	7
Sr	-	-	-
Ti	-	-	0.2
Zn	-	5	-
Zr	-	-	-

<sup>a</sup> ±50% of actual values. Dashes indicate none found.

<sup>b</sup> Styrene Plastic Crystal, lot No. 11, SMD-5601. Union Carbide and Carbon Corp., Bakelite Division, 30-E 42nd Street, New York, New York.

<sup>c</sup> Nylon backing and facing removed after pads had been rolled. Special precautions observed to maintain cleanliness of pad throughout processing. Pads prepared June 10, 1959.

A typical photomicrograph, taken with a framing camera as described in "d" of the preceding section, is shown in Figure 6. If the fibers resulted from simple elongation of the initial jet of solution by air blast, the air blast velocity would have to be several orders of magnitude greater than the velocity of sound to maintain the rate of production which was achieved. To obtain very fine fibers, the solution must be sufficiently viscous to prevent the newly formed fibers of solution from collapsing into droplets, but not so viscous that the air blast is prevented from making the fine solution fibers in the first place.

The filters probably collected particles in the size range of interest largely by two mechanisms, namely, direct interception and diffusion. The former mechanism was probably most effective for particles larger than about  $0.5 \mu$ , and the latter for particles smaller than about  $0.1 \mu$ . Thus, as has been discussed by many authors (7), one might expect to find a minimum in the curve of particle size vs collection efficiency. Such a minimum does occur, as is demonstrated by the results shown in Table II.

A comparison of filter performance on the basis of collection efficiency alone can be misleading, since it does not consider other properties of interest such as pressure drop. A better basis is the use of a parameter,  $F$ , which is defined by the differential equation

$$100 \frac{dc}{c} = -F dp$$

where  $c$  is the number concentration of particles and  $p$  is the pressure at any point in the filter.  $F$  is readily calculated from the integrated form of the equation, namely,

$$\frac{c_1}{c_0} = \exp \frac{-F\Delta P}{100}$$

where  $c_1$  is the number concentration of particles leaving the filter and  $P$  is the pressure drop across the filter. This parameter is particularly useful for comparing various filters at a given face velocity. Thus the performance of the filters described in this paper can be compared directly with those studied by Wentz (8) and by Wright, Stasny, and Lapple (9).



FIG. 6  
PHOTOMICROGRAPH OF FIBERS FORMING FROM THE  
SOLUTION LEAVING THE NOZZLE

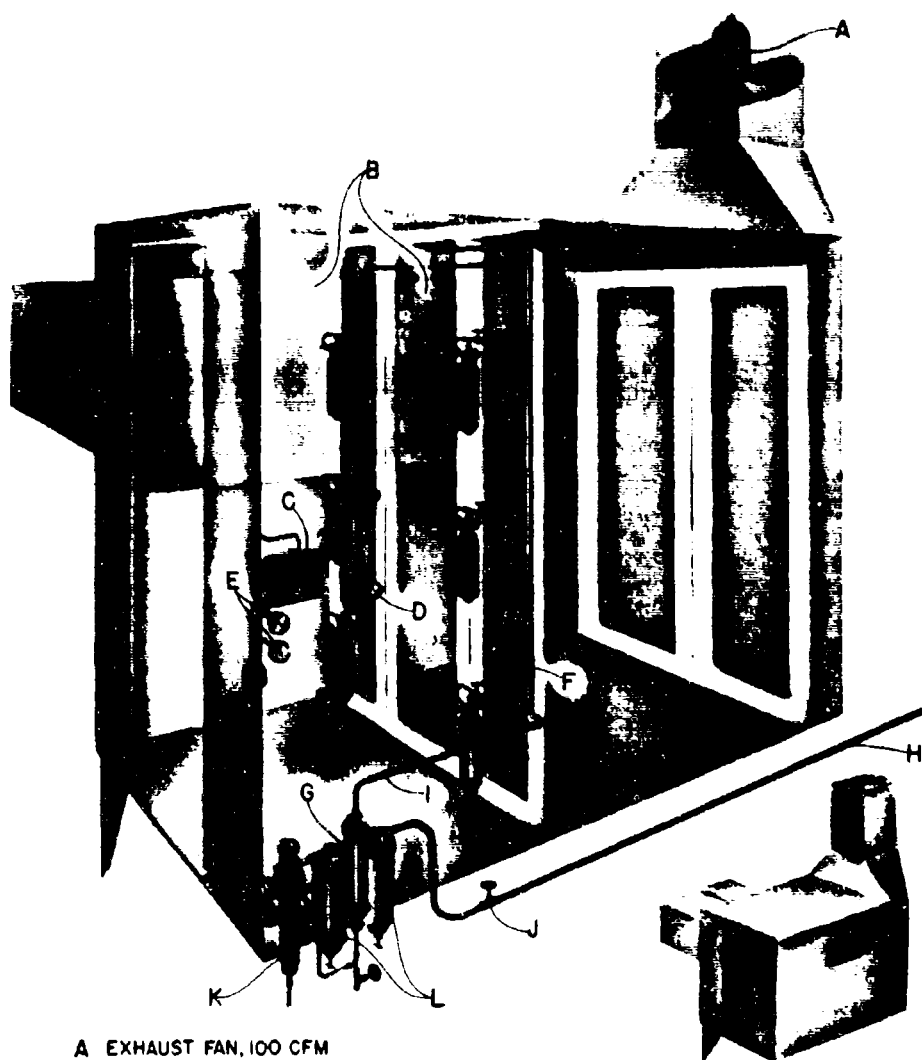
Values of F are shown in Tables I and II. Compressing the filters decreased their performance considerably, as would be expected. The performance of the compressed polystyrene filters was approximately that of the German filters, the CWS#6 filter, and the organic fiber filters prepared by Wentz. The high performance of the filters prepared from 2- $\mu$ -diameter fibers compared with those prepared from smaller fibers is of interest, and results from the low pressure drop produced by the larger fibers.

The effect of low atmospheric pressures, such as the pressure in the stratosphere, on collection efficiency was an important consideration. One example of this effect was calculated by using the mathematical treatment of filter performance given in the publication of Wright, Stasny, and Lapple (9). Collection efficiencies were calculated for 1.0, 0.1, and 0.01 atmosphere, using a constant face velocity through the filter and representative properties and dimensions for the filter and aerosol. Stated in general terms, the calculation showed that the filter collection efficiencies of both diffusional and inertial mechanisms were greatly enhanced by decreasing the atmospheric pressure.

#### E. Pilot Plant Facilities, Operation, and Product Testing

##### 1. Pilot Plant Facilities

A drawing of the chamber in which the 3,000 sq ft of filter were processed is given in Figure 7. Dimensions of the chamber are given in Figure 8. It measured 8 ft, 5 in. by 6 ft by 7 ft in height and was made of galvanized iron. Two sections of filter pad, each finally cut to the required 24 in. by 70 in., were prepared simultaneously. The fibers for each section were produced by 3 generators which were mounted one above the other, 4-1/2 ft from the collecting areas. The generators were used to spray a solution of 10 g of polystyrene per 100 ml of methylene chloride into the collecting areas, and have been described previously. A single generator is shown in detail in Figure 1, and the banks of generators and the two collecting areas are shown in Figures 7 and 9. The banks of generators were connected to the air line as shown in Figure 7 by means of



- A EXHAUST FAN, 100 CFM
- B CAMBRIDGE "ABSOLUTE" FILTERS, INTERIOR FACE BAFFLED
- C CHAMBER PRESSURE INDICATOR
- D AIR PRESSURE GAUGE
- E HYGROMETER
- F COMPRESSED AIR VALVE
- G FILTER COLUMN, GLASS WOOL PACK, VOLUME, 250 cc
- H COMPRESSED AIR LINE
- I COPPER TUBING 3/8 IN OD
- J COMPRESSED AIR VALVE
- K SPRAYIT MODEL 910 AIR FILTER AND REGULATOR
- L FULFLO MODEL AFB AIR FILTERS

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FIG. 7

CHAMBER USED FOR PREPARING POLYSTYRENE FIBER FILTER PADS, 70 IN. BY 24 IN.

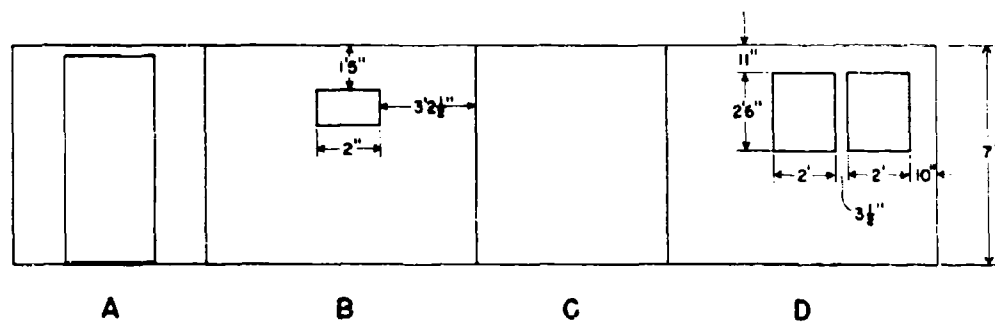
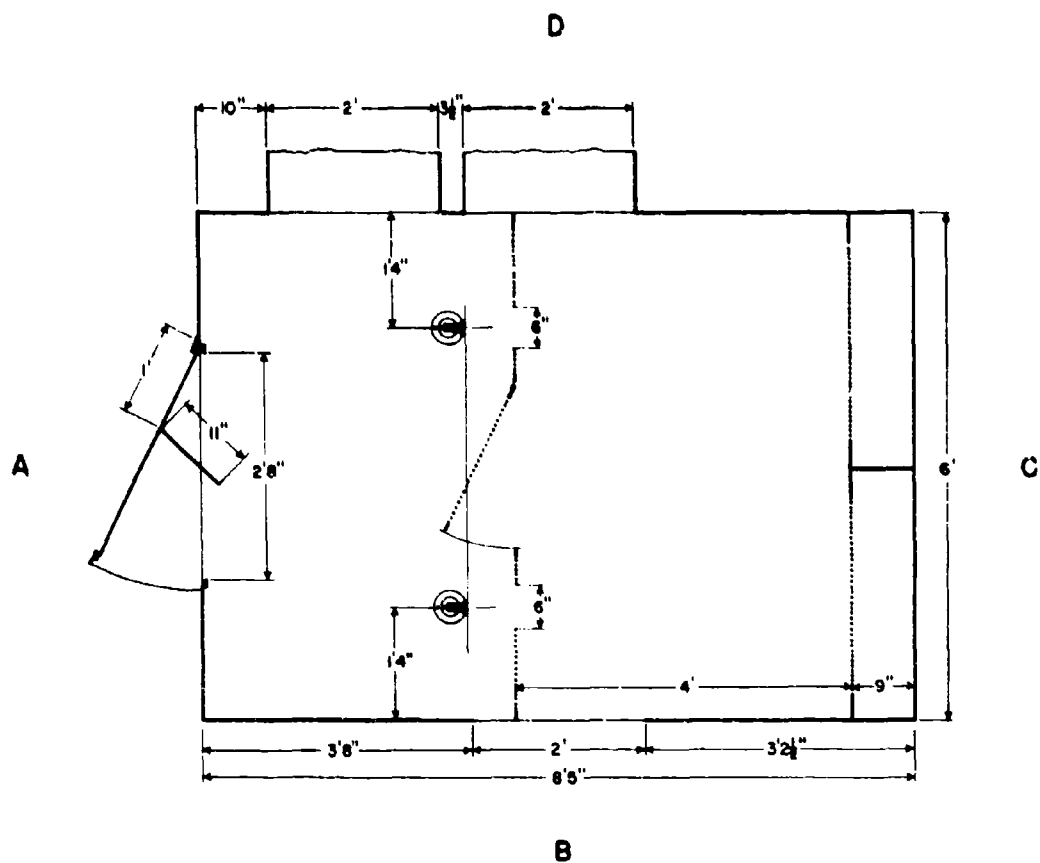


FIG. 8  
DIMENSIONS OF FILTER PROCESSING CHAMBER



FIG. 9

CHAMBER INTERIOR, SHOWING TWO COLLECTING AREAS AND TWO BANKS OF GENERATORS



3/8-in.-I.D. copper tubing. An air pressure of 30 psi was supplied to the 6 generators from a 10-hp compressor.

The system for cleaning the compressed air is also shown in Figure 7. Before entering the generators, air from the compressor was filtered through 2 "Fulflo," Model AFB, compressed air filters connected in series,\* then through a "Sprayit," Model 910 air filter and regulator,\*\* and finally through a brass pipe, 2.5-in. O.D., 10 in. long, fitted with glass wool. All the filters were drained daily. No contamination of the glass wool, the last filter in line, was observed in the course of preparing the 3,000 sq ft of filter material.

The collecting areas, 72 in. by 26 in., were constructed at one end of the chamber, 9 in. in front of the chamber wall. They were covered with wire screen over which 32-mesh Dacron netting was stretched for collecting the fibers, as shown in Figure 10. Air was drawn through the collecting areas and vented to the outside by means of a 100-cfm exhaust fan, shown in Figure 7. Two Cambridge "absolute" filters,\*\*\* installed in parallel in one wall of the chamber, were used to filter the outdoor air which was drawn through the collecting screens during operation. "Dust Stop" fiberglass filters\*\*\*\* were used as pre-filters for the air entering the Cambridge filters, in order to increase their lifetime. The Dust Stop Filters were changed weekly. Baffles were mounted in the chamber in order to circulate the incoming air so that a uniform distribution of fibers was obtained on the collecting areas. The positions of the baffles and "absolute" filters are shown in Figure 7. The door to the chamber was made to fit tightly so that there would be no contamination from unfiltered air during filter fabrication. A pressure drop of 0.3 in. of water was obtained across the "absolute" filters during operation, as

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\*Commercial Filters Corporation, Melrose 70, Massachusetts.

\*\*Thomas Industries Inc., Electric Sprayit Division, Sheboygan, Wisconsin.

\*\*\*Cambridge Filter Corporation, P.O. Box 1255, Syracuse 1, New York.

\*\*\*\*Dust Stop Type B Fiberglass Air Filter. Lynch Asbestos Company, 2939 Sunol Drive, Los Angeles, California.



FIG. 10  
COLLECTING AREA BEING COVERED WITH DACRON NET

indicated by the manometer shown in Figure 7. The humidity of the circulating air was measured daily by means of wet- and dry-bulb thermometers, also shown in Figure 7. The relative humidity varied from 30% to 50% during the period of filter fabrication (May to September, 1959), and the temperature varied from 65°F to 80°F. These are normal values during this period at Menlo Park, California.

A screen partition was located immediately in front of the generators, and is shown in Figures 7, 8, and 9. Openings, 8 in. by 8 in., were cut in the screen in front of each generator nozzle. This screen as well as the direction of air movement in the chamber prevented most of the drifting excess fibers from reaching the operator, who was located in the compartment containing the generators. Solvent odor could not be detected from the position of the operator. There was a door in the screen for access to the front of the chamber.

It was important to develop a procedure for keeping the processing chamber as clean as possible to prevent contamination of air in contact with chamber surfaces. The chamber (including the floor) was cleaned initially with distilled water, dried, polished with clean cloths, and sprayed with 2 coats of Krylon Clear coating. Shoes were not worn in the chamber, and the filters were handled with nylon gloves. The chamber was kept closed tightly, and was cleaned with a vacuum cleaner daily.

The mats which were prepared in the chamber were transferred to a dust-free room, 18 ft by 12 ft, for final processing and packaging. The room contained a 3 by 11-ft work table covered with plate glass. Mats were rolled, as described below, with a 35-lb glass roller, filled with shot, which was 40 in. long and 2 in. in diameter. Surfaces in contact with the filters were cleaned daily.

## 2. Pilot Plant Operation

Sets of 2 filters, 24 in. by 70 in., were prepared by the following method:

- a. The polystyrene solution (10 g of polystyrene per 100 ml of methylene chloride) was prepared by transferring 1500 g of polystyrene to a clean glass container and adding 15 liters of methylene chloride.\* About 24 hr were required to dissolve the polystyrene, and a second container of solution was kept on hand. The containers were kept dust free and were equipped with glass siphons.
- b. Polystyrene solution, 350 ml, was transferred to each of the 6 fiber generators. The generators were fastened in place.
- c. The collecting areas were covered with Dacron nets, 24 in. by 72 in., as shown in Figure 10. The nets were kept as wrinkle free as possible and were held in place with hooks in the edges of the collecting areas.
- d. The main air valve in the chamber (F in Figure 7) was closed. The compressed air was turned on, and the pressure regulator (K in Figure 7) was adjusted to 70 psi.
- e. The operator entered the chamber, closed the door tightly, opened the 6 air valves of the generators, turned on the exhaust fan, and turned the adjusting screws located at the rear of each generating nozzle (Figure 1) so that the liquid-jet orifice was closed.
- f. The main air valve in the chamber (F in Figure 7) was opened until air escaped from the 6 nozzles under a pressure of 50 psi as noted on the gauge (D in Figure 7).
- g. The liquid-jet orifice of each generator was opened in turn. The extent to which the orifice was opened varied somewhat with each generator, and was determined by observing, for the production of fine, discrete, dry fibers in a beam of light located about 20 in. downstream of the nozzle. At the correct opening, no liquid, and no odor of methylene chloride, was obtained on a cardboard target held in this region, and 60 to 75 min were required to form a set of pads from the full 350 ml of solution in each generator. Many hours were required to form a set of pads if smaller liquid orifices were used. If the orifices were opened too far, the pads were irregular, and contained areas dissolved out by methylene chloride which had not had time to evaporate in the air stream. It was necessary to re-adjust the orifice openings slightly in the course of a run in order to build up uniform pads. A few days' practice

\*Granular polystyrene was obtained from The Union Carbide and Carbon Corporation, Bakelite Division, 30 East 42nd Street, New York City. The material was labelled Styrene Plastic Crystal 11, SMD-5601. The methylene chloride (CP) was obtained from Braun-Knecht-Heimann Company, San Francisco, California.

were required in manipulating the liquid jets for an operator to achieve facility in obtaining uniform, reproducible pads, and to discharge the polystyrene solution from the six generators at an equal rate.

- h. About 50% of the fibers did not reach the Dacron nets. A portion of the fibers collected in large bundles extending out from the edges of the pads, and were kept from falling onto the pad surface by the circulating air. Another portion of the fibers was drawn together while free-floating due to the electrostatic properties of the material, and accumulated on the floor of the chamber. When the solution in the generators had been discharged, the main air valve in the chamber was turned off and the bundles of fibers along the edges of the pads were removed by hand. The exhaust fan was then turned off.
- i. The exposed faces were covered with Dacron netting before the pads were removed from the chamber. Wooden frames, the size of the collecting areas and covered with Dacron nets, were placed over the pads with the Dacron in contact with the pad surfaces. The Dacron nets on which the fibers had been collected were then fastened to the frames, forming "sandwiches" of fibers between Dacron. The pads, supported by the frames, were then carried to the dust-free room. Pads, with and without the frame in place, are shown in Figure 11.
- j. The frame was placed on the glass-top table with the pad on the glass. The frame was then carefully removed, and the edges of both layers of Dacron were fastened to the glass under moderate tension to remove creases. The pad was then compressed by rolling 4 times with the glass roller, as shown in Figure 12.
- k. A frame, 24 in. by 70 in., was centered on top of the compressed pad, and light pencil lines were drawn on the pad around the boundaries of the frame. The frame was then removed, and the pad was cut to size with scissors. A completed filter pad is shown in Figure 13.
- l. The Dacron scrim would detach from the layer of fibers if the filters were rolled up, so they were stored and shipped flat. Lots of 10 filters were stored in polyethylene envelopes. The envelopes were stapled into crates to prevent movement during shipment.

### 3. Product Testing

Five tests of collection efficiency, pressure drop, density, and purity of filters were made at intervals during the pilot plant operation, which

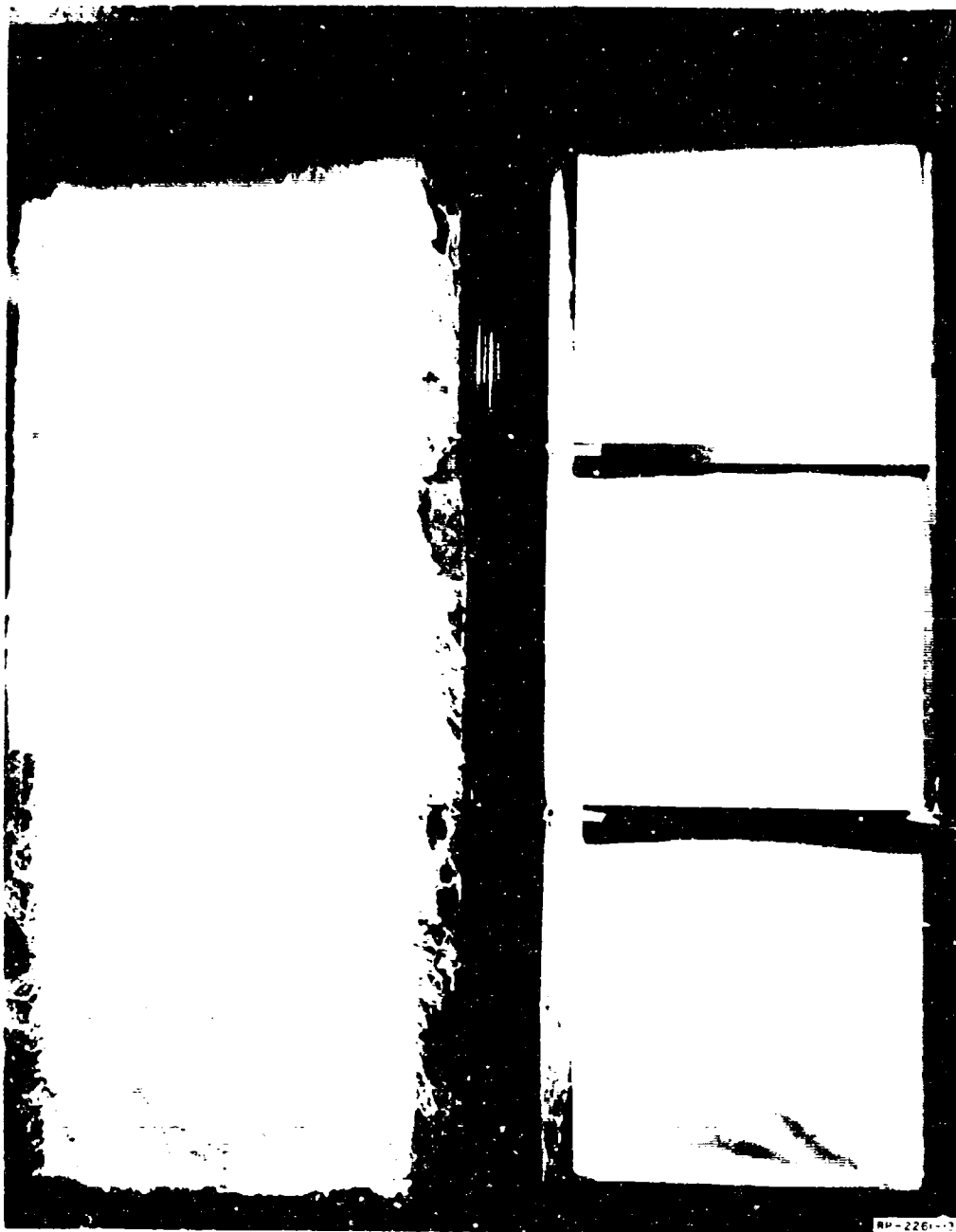


FIG. 11

LEFT: APPEARANCE OF PAD AFTER FIBER COLLECTION  
RIGHT: PAD WITH DACRON-COVERED FRAME IN PLACE



FIG. 12  
COMPRESSION OF PAD BY ROLLING



FIG. 13  
COMPLETED FILTER PAD



extended from May to September, 1959. Pads to be tested were selected at random from a week's supply. The thinnest region of the pad was tested for collection efficiency, and the thinnest and thickest regions for pressure drop. These tests were made with the Dacron in place. The fibers of the entire pad were then collected in bulk and weighed without the Dacron, in order to obtain the average density per sq ft of fiber material. The fibers of other pads were collected in bulk, also without the Dacron, and were analyzed for trace metals by emission spectrography.

The results of the control tests of collection efficiency, pressure drop, and density are given in Table IV. Lithium fluoride aerosols with particles  $0.1 \mu$  in diameter were used in the efficiency tests. The flow rate through the filter was 14 cm/sec. As was discussed previously, a minimum in collection efficiency was obtained with particles of this size over the range  $0.01 \mu$  to  $1.0 \mu$  diameter. The collection efficiencies ranged from 99.5 to 99.9 for the  $0.1\text{-}\mu$  particles. Higher collection efficiencies would be obtained for particles in the lower and upper size ranges, and in tests of thicker regions in the filter. The pressure drop across the test filters was also measured at a flow of 14 cm/sec. It ranged from 92 to 115 mm of water in tests of the thinnest and thickest regions of the filters. The average density of the fiber material varied from 3.7 to 4.3 g/sq ft.

The emission spectrographic analyses of 5 filters are given in Table V. Metallic contamination in the fibers ranged from about 0.1 to 10 ppm by weight, with only calcium and silicon in the upper range.

Table IV

TESTS OF COLLECTION EFFICIENCY, PRESSURE DROP, AND DENSITY OF FILTERS  
MADE DURING PILOT PLANT OPERATION

Date of Filter Preparation	Collection Efficiency, %, for Lithium Fluoride Particles, 0.1 $\mu$ Diameter <sup>a</sup>	Pressure Drop Across Filter, mm H <sub>2</sub> O <sup>a</sup>		Average Density of Fiber Material, g/sq ft <sup>b</sup>
	<u>Thinnest Region</u>	<u>Thinnest Region</u>	<u>Thickest Region</u>	
5/25/59	99.9	102	115	4.3
7/6/59	99.5	97	111	3.8
7/24/59	99.8	85	100	4.2
8/17/59	99.7	98	105	3.7
9/11/59	99.8	92	103	3.8

<sup>a</sup> Collection efficiency and pressure drop across filter measured at 14 cm/sec linear flow.

<sup>b</sup> Both layers Dacron removed.

Table V  
CONCENTRATION OF TRACE METALS IN FILTERS MADE DURING PILOT PLANT OPERATION  
Analysis by Emission Spectrography  
(ppm by weight)

	Pad Prepared June 10	Pad Prepared July 1	Pad Prepared July 20	Pad Prepared July 31	Pad Prepared August 17
Al	2	2	5	3	1.5
Ba	-	0.3	0.2	0.5	0.2
Ca	10	5	11	6	10
Cr	0.04	0.3	0.1	0.1	0.1
Cu	0.1	0.2	0.3	0.4	0.4
Fe	1.0	1.5	2.3	5.9	1.0
Mg	0.7	5	2	5	5
Mn	0.07	0.3	0.03	0.12	0.05
Na	-	1.3	2	3	7
Ni	-	0.3	0.1	0.1	1.1
Pb	-	0.5	0.6	0.6	0.4
Si	7	10	6	3	3
Sr	-	0.3	0.1	0.1	0.1
Ti	0.2	2.7	0.1	1.2	1.2
Zn	-	7	-	-	6
Zr	-	-	-	-	0.1

Nylon backing and facing removed after pads had been rolled. Reported values  $\pm 50\%$  of actual values. Dashes indicate none found.

## Part II

### A SURVEY OF SOURCES OF ORGANIC FILTER MATERIAL APPLICABLE TO ATMOSPHERIC SAMPLING

#### A. Introduction

At the beginning of the project in August, 1957, a trip was taken to various fiber manufacturers in the U.S. to determine whether any currently available filter material would meet the specifications required by Air Force Cambridge Research Center. These specifications are given in the Introduction to Part I of this report. Although no suitable filter was found, and the method described in Part I was developed to fulfill the special requirements, the survey of sources of available filter material was continued during the development of the process and the final fabrication of the filters in order to obtain the following information: (1) an understanding of the "state of the art," present developments, and future potential of organic filter manufacture, (2) methods to be investigated for improving our filter, (3) methods of processing organic fibers into filter papers, (4) an evaluation of the interest of certain manufacturers in producing small quantities of special filter material for Air Force Cambridge on a contract basis and (5) patent coverage of spray methods for producing organic fiber filters. Most of the information was obtained by means of visits with company representatives in the U.S., Britain, Germany, and Italy. Twenty-five organizations were included. This, of course, by no means represents a survey of the man-made fibers industry, as the industry includes some 80 plants in the U.S. alone (10). The survey does, however, include the leading sources of organic filter material available on laboratory, pilot plant, and commercial scales. For convenience, organizations are divided into these three categories in the survey.

#### B. Summary

Significant information was obtained on the processing of organic filters, together with an understanding of the present state of the art of

organic filter production, and of the future potential in this field. Information concerning possible methods of improving the filter described in Part I of this report included (1) processes developed by American Viscose Corporation for spraying the plastic through an electrostatic field to prevent fiber bundling and to increase fiber recovery, (2) suggestions from W. and R. Balston and the Hollingsworth and Vose Company concerning the advantage of a distribution of fiber diameters to increase filter strength, (3) processes developed by the Chemical Defence Experimental Establishment in England for strengthening the fiber filters by heat-bonding, (4) suggestions from the Millipore Filter Corporation on methods of softening the fibers just enough to cause bonding at contacts, and (5) the use of Kimberly-Clark paper consisting of Dacron or Nylon fibers as a substitute for the Dacron mesh used to re-enforce our polystyrene fiber mat.

Information on methods of processing fibers into papers was obtained from the following organizations: (1) Armour Research Foundation, (2) Arthur D. Little, Inc., (3) Cambridge Filter Corporation, (4) Dow Chemical Company (through the U.S. Army Chemical Warfare Laboratories), (5) Hollingsworth and Vose Company, (6) Mine Safety Appliances Company, and (7) Schleicher and Schull Company. It was concluded that paper-making techniques were generally impractical for processing fine-fiber organic filters such as those described in Part I of this report.

The following organizations indicated a varying capability and interest in producing pure, organic filters with specified characteristics, and in relatively small quantities: (1) Arthur D. Little, Inc. (if paper-making processes could be used), (2) W. and R. Balston, Ltd. (who have recently developed a filter very similar to the one described in Part I of this report), (3) Membranfilter gesellschaft (if polystyrene membrane filters were applicable), (4) The Millipore Filter Corporation (if the development of high-purity membrane filters could be sponsored), and (5) Delbag-Luftfilter, who are presently marketing a filter very similar to the filter described in Part I of this report

Information on patent coverage of spray methods for producing organic fiber filters was obtained from the following organizations: (1) American Viscose Corporation, (2) Delbag-Luftfilter (U.S. patents applied for), and (3) Chemical Defence Experimental Establishment at Porton (U.S. patents applied for). Information on patent coverage is by no means complete in this survey, and it is recommended that the patent status of the organizations listed above be investigated and that licenses be obtained if necessary, before a spray process is used to provide filters on a contract basis.\*

C. Producers of Organic Filter Material on a Laboratory Scale

1. Armour Research Foundation of Illinois Institute of Technology  
(Technology Center, 10 West 35th Street, Chicago 16, Illinois)

During the period 1948-1950, Armour Research Foundation conducted an investigation with the objective of developing a low ash filter which would have a low pressure drop, have a high collection efficiency for particles 1  $\mu$  in diameter, and be soluble in an organic solvent.

Three fibers were studied: polystyrene, Vinyon, and cellulose acetate. Resin binders of polystyrene, polyisobutylene, and polyvinyl butyrate were utilized. The cellulose acetate and Vinyon fibers were obtained from commercial sources. The original polystyrene fiber was obtained from a small sample available at the Dow Chemical Company, which had two machines for making very fine fibers. Dow later disposed of both of these machines and one was acquired by the U.S. Army Chemical Warfare Laboratories. The work at both the Dow Chemical Company and the Army Chemical Center is described later in the survey.

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\*The following patent should also be considered: U.S. Patent No. 2,713,001, July 12, 1955, by F. W. Manning. It concerns a method of wrapping the carcasses of freshly killed animals by coating them with a fabric of fibers produced by a spray method. Claim 1 of 28 claims is as follows: "An integral nonwoven fabric comprising a plurality of discontinuous filaments extending beyond one another in substantially uniform overlaps and bonded to one another."

The process used at Armour Research Foundation to fabricate the filter involved use of standard paper-making equipment, with reasonable precautions to avoid contamination. The cellulose acetate or Vinyon fiber was obtained as a floc, 2-6 mm long, straight or with a crimp, and of the lowest denier possible. This was dispersed in water with a conventional paper beater, and diluted to make a suspension. The suspension contained of the order of 2 lb of fibers in 300 gal of water (0.08% fibers). While this suspension was gently and uniformly agitated, a screen suction box was lowered into the suspension. Water was removed through this box and the filter pad formed on the screen. In some cases the screen was covered with an open mesh cellulose acetate cloth to support the fragile pad before impregnation. The pads were oven-dried. Impregnation was accomplished by dipping the pad in an impregnating solution containing 1 to 4% binder, and drawing off the excess material on a suction box. The remaining solvent was removed by either air-drying or oven-drying.

Collection efficiencies up to 97 wt % were obtained when testing aerosols of carbonyl iron particles with diameters up to 3  $\mu$ . Flow rates of 5.5 fps at 10 in. of water were used. The filters weighed about 15 g/sq ft, and were about 0.020 in. thick. They could not withstand rough handling, but could easily be stacked for storage or transferred in filter holders.

Limited tests with air felting techniques were made. Considerable difficulty was encountered in obtaining uniform dispersion of the fibers.

This information was obtained from M. A. Fisher, Supervisor, Fine Particles Research, Armour Research Foundation.

2. Arthur D. Little, Inc. (Acorn Park, Cambridge 40, Massachusetts)

One of the objectives of the Arthur D. Little Corporation in its studies of filters has been to develop high efficiency industrial filters, such as the Aerosolve and Absolute filters developed for the AEC (11). These filters are manufactured by the Cambridge Filter Corporation. The developments at Cambridge Filter Corporation are described later.

The studies at Arthur D. Little, Inc. indicated that very fine synthetic fibers can be wet-formed into very effective air-filter media. Their most successful air-assay papers were made from synthetic fibers combined with cellulose fibers. The sheet could be acid-treated to produce a low-ash paper. Paper-making techniques were used to prepare sheets with fibers of cellulose acetate, Vinyon, Nylon, Dacron, Dynel, and acrylonitrile. Their best results were obtained with acrylonitrile, which is readily obtainable in fine-diameter sizes, and which disperses readily in aqueous slurries, using Dahad No. 11 as the dispersing agent (11). A paper prepared by mixing acrylonitrile fiber, cotton linter fiber, Daxad, and water in a blender had the following performance when tested at 28 fpm with DOP (diocylphthalate) smoke:

smoke penetration - 0.32%

pressure drop - 240 mm H<sub>2</sub>O.

It was observed at Arthur D. Little, Inc. that a glossy filter surface may be unsatisfactory when used in heavily polluted atmospheres because of surface buildup of the filtered material, but that in cases of continuous sampling of the upper atmosphere the collection capacity of the filter would probably remain unchanged for weeks.

If paper-making processes could be used to make filter material on a limited scale, Arthur D. Little, Inc. could manufacture the material by using its paper-making crew and paying a local paper firm for the use of its equipment.

Arthur D. Little, Inc. has also investigated the production of microfibers from organic plastics by spray techniques for the American Viscose Corporation. Results of these investigations are described in the section on American Viscose Corporation later in the report.

The information was obtained from Dr. Howard O. McMahon and Dr. Walter J. Smith of Arthur D. Little, Inc.



3. Naval Research Laboratory (Washington 25, D.C.)

The Naval Research Laboratory has developed a process for producing fibers of thermoplastic materials with diameters down to 0.1 micron, using an extrusion process (12). No particular attention was given to the purity of the resulting material, and the metallic content was too high for the present application. However, the process would be useful if it was necessary to produce fibers from specially purified thermoplastic materials, including Nylon, polystyrene, polymethyl methacrylate, polyethylene terephthalate, and polytrifluorochloroethylene.

The apparatus developed for the production of fine fibers is essentially a ram-extruder that forces molten material through a row of fine orifices and directly into two converging high velocity streams of heated air. The air blast is separated from the fiber stream by passing through a 16-mesh collecting screen which moves across a suction chamber. A random network of fibers is deposited on the screen and then stripped off to be wound onto a reel either with or without being densified by pressing. The process is described in detail in reference 12.

The information was obtained from Dr. W. L. Anderson of the Naval Research Laboratories and from the paper of Van A. Wente, cited above.

4. U.S. Army Chemical Warfare Laboratories (Army Chemical Center, Maryland)

The Army Chemical Center had a project in 1950 to develop an organic filter for the Air Force (AFOAT). The filter was to be used for upper air sampling during atomic tests, using drone planes to carry the filter through the cloud. The filter was to have essentially a zero ash content. The Dow "Polyfiber" process was used. The Dow Chemical Company had two machines for making very fine fibers, and one was acquired by the Army Chemical Center for its investigations. A solution of polystyrene in cumene (a substituted benzene) was poured between two very close, parallel cylinders which rotated in opposite directions. The polystyrene pulled away from the surfaces to form fibers. The fibers were then

scraped away and were formed into a mat. Fibers with diameters less than  $1\ \mu$  could be produced by this method. A tendency of the fibers to pull away from the mat surface was prevented by spraying the mat with a solution of polyvinyl butyral in alcohol. The surface was made tacky by adding polyisobutylene. The filter was evaluated with D.O.P. aerosol in which the droplets were  $0.3\ \mu$  in diameter. The pressure drop through the filter was 8-10 mm water, with a face velocity of 2400 ft/min. The filter collected the D.O.P. particles with an efficiency of 99%. Some of the material was supplied to the Air Force, and the pilot plant was subsequently dismantled. A complete description of the Dow process is given in A. W. Hanson Patent No. U.S. 2,417,751.

The Chemical Corps has developed a combination aerosol-gas filter by combining fibers and finely divided charcoal, using a wet accretion method. The filter is discussed under Mine Safety Appliances Corporation, below.

This information was obtained from Mr. Jack Fleming and Mr. Bert Karpel, Deputy Chief, Protective Development Division, U.S. Army Chemical Warfare Laboratories.

D. Producers of Organic Filter Material on a Pilot Plant Scale

1. The Bakelite Company, Division of Union Carbide (Bound Brook, New Jersey)

The Bakelite Company has developed a polystyrene and vinyl polymer fiber material produced by an undisclosed process. The material has been produced in multi-thousand pound lots, but is not on the market. The fibers are coarse, but very fine fibers can be prepared. The fibers can be combined with wood pulp and made into paper, using standard paper-making equipment, or filters can be prepared from the fibers alone. The Bakelite Company is interested in finding applications for the product.

The information was obtained from Dr. F. M. Rugg, Assistant Director of Research for The Bakelite Company.

2. W. and R. Balston, Ltd. (Maidstone, Kent; England)

W. and R. Balston, Ltd. is a paper manufacturer, and Whatman filter paper is one of its products. It has developed a process for producing organic fiber filters which is adaptable to small-scale manufacture, using an extrusion apparatus similar to that developed at the Naval Research Laboratory, described above. It is developing another, undisclosed, extrusion method.

The W. and R. Balston staff did not recommend paper-making techniques for making organic fiber filters because the process would be relatively complex, and the filter strength possibly wouldn't be increased very much. It was their opinion that carding methods would be unlikely to give uniformity in strength and filter efficiency, and that although a mixture of fiber sizes would increase the tearing strength, it would not increase the pulling strength, i.e., the resistance of the pad to being pulled apart sufficiently to damage any area of the pad without actually tearing it. A process of actually bonding the fibers would be necessary to increase the pulling strength.

The W. and R. Balston Company would be interested in preparing pure organic fiber filters in limited quantities. They said that they could reach the AFCRC standards for the present filter very quickly, since the new extrusion method which they are investigating produces a filter with about the same characteristics as the SRI filter described in Part I. In August 1959, they reported that they had developed a device for producing small quantities of fiber filter, using the method described in Part I of this report with certain modifications. They expected to have a system capable of producing larger quantities of the material by a continuous method in operation later in 1959.\*

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\*A sample of Balston fiber filter paper was received at Stanford Research Institute in September, 1959. It appeared to have the texture and strength of the SRI material. The filter had a pressure drop of 50 mm water and a collection efficiency of 99.3% for 0.1- $\mu$ -diameter particles when tested at a flow rate of 14 cm/sec through the filter. The material was not analyzed for impurities, but otherwise appeared as satisfactory for the present purpose as the material described in Part I of this report.

The information on W. and R. Balston, Ltd. was obtained from Mr. John Balston, Director of Research, Mr. B. M. Thomas, Production Engineer, and Mr. B. E. Talbot and Mr. A. H. Pooley of the Engineering Department.

3. The Dow Chemical Company (Midland, Michigan)

The Dow Chemical Company has produced a polystyrene fiber, "Polyfiber," with fiber diameters from 1 to 8  $\mu$  and a median diameter of 3  $\mu$ . The process for making Polyfiber is described above, under U.S. Army Chemical Warfare Laboratories. The process has been discontinued, and the machinery dismantled.

The information on Dow Chemical Company was obtained from Dr. P. H. Lipke, Manager, Testing and Special Product Development, Plastics Technical Service.

4. Kimberly-Clark Corporation (Neenah, Wisconsin)

The Kimberly-Clark Corporation has made both Dacron and Nylon sheets in a broad range of weights, ranging from 7 lb/3000 sq ft to about 75 lb/3000 sq ft. For the light weight sheets they used fibers of 1.5 and 3.0 denier (about 14  $\mu$  and 19  $\mu$  in diameter, respectively). Coarser fibers are available and have been used in some of their heavier sheets. The sheets consist of about 75% fiber and 25% thermoplastic binder melting at about 400°F. The sheets that are produced in very light weight are open, net-like structures and will pass large volumes of liquid or gas at low pressure differentials. The sheets are available in widths up to about 30 in. Prices are not yet established, but they estimate a price of about 1¢/sq yd/lb of basic weight. Thus, sheets weighing 10 lb/3000 sq ft would be priced at about 10¢/sq yd.

The sheets are strong enough to serve as a substitute for the Dacron net which has been used to re-enforce the filter described in Part I of the report, and the pressure drop across the thin sheets would be negligible. However, no data were available on the filter efficiency of

the material. Preliminary tests were run at Stanford Research Institute on a sample received from Kimberly-Clark. The sheet was made of Nylon, and weighed 30 lb/3000 sq ft (Kimberly-Clark Run No. SF64, Type 043030F). The results are given in Table I, below.

Table I  
PRESSURE DROP AND COLLECTION EFFICIENCY OF KIMBERLY-CLARK  
SYNTHETIC FIBER PAPER, TYPE 043030F

Number of Thicknesses	Collection Efficiency, %*	F (mm <sup>-1</sup> )	p, mm H <sub>2</sub> O**
1	10	5.0	2
2	20	5.5	4
4	33	5.0	8
8	50	4.3	16
Extra- plicated 50	~95	~3.0	100

\*Percent retention of lithium fluoride particles 0.07  $\mu$  in diameter, at flow rate of 28 ft/min through paper.

\*\*Flow rate through paper, 28 ft/min.

Single sheets of the material have a collection efficiency of about 10% for particles in this size range (0.07- $\mu$ -diameter), which is too high to consider as reinforcement for the present polystyrene fiber filter material if quantitative collection on the polystyrene fiber mat is required. A thinner sheet, made of Dacron and weighing 10 lb/3000 sq ft, was also tested (Kimberly-Clark Run No. SF56, Type 099010F). The pressure drop across a single sheet of this paper under the above test conditions was less than 1 mm H<sub>2</sub>O, and the collection efficiency was less than 1% for the 0.07- $\mu$ -diameter lithium fluoride particles. Because of these low values it was not possible to carry out further preliminary tests. This material could possibly be used as a reinforcement for the polystyrene material, but tests of collection efficiency for particles in the 1.0- $\mu$ -diameter range would be required, with consideration given to the increase in filter efficiency of the paper at high altitudes.

It is interesting to note (Table I) that very high filter efficiency would be expected with 50 thicknesses of the filter tested, and that the pressure drop would be only 100 mm H<sub>2</sub>O.

The information was obtained through correspondence with Mr. Paul B. Hansen, of Project Team-New Products, Corporate Product Planning, at the Kimberly-Clark Corporation.

E. Producers of Organic Filter Material on a Commercial Scale

1. American Viscose Corporation (1617 Pennsylvania Boulevard, Philadelphia 3, Pennsylvania)

The American Viscose Corporation has been investigating methods of production of synthetic fibers by spray techniques for several decades. In general, the process, apparatus, and microfiber products\* are covered by claims of issued patents owned outright by American Viscose.

The general process of fiber formation by spraying is described in Carleton S. Francis patent 2,483,404 of October 4, 1949. This patent is a continuation-in-part of Francis patent 300,876, filed October 23, 1939, now U.S. Patent No. 2,459,803, and 381,292, filed March 1, 1941, now U.S. Patent No. 2,357,392. The fiber-forming material is dispersed while in solution into a gaseous atmosphere under sufficient pressure to form a multiplicity of fibers, using a spray gun or atomizer. Air is supplied from the region adjacent to the orifice through which the solution is forced under pressure. The air disperses and disrupts the stream of fluid issuing from the orifice, forming fibers. The fibers may be collected on a retaining wall to form an integral, pervious web. The web of fibers may be made adhesive in order to bind some of the fibers together, and if desired, to bind the web to other materials. The use of sprayed fibers in the manufacture of paper is also covered by generic claims in patent No. 2,357,392.

\*The term microfibers as it relates to patents of the American Viscose Corporation is defined as follows: "The extremely fine, irregular dimensional plastic fibers formed by disrupting a plastic either in molten condition or in solution, into an air stream."

The process, apparatus, and microfiber products are described further in Francis patents, U.S. patents No. 2,483,405 and 2,483,406, both of October 4, 1949. The latter patent also covers a composite product in which rubber fibers are deposited as a distinct layer upon another layer of fibrous material.

Broad claims directed to the spraying of plastic fibers through an electrostatic field are contained in patent No. 2,810,426, of October 22, 1957, by Derek E. Till and Carl R. Smallman. The work is also described in a monograph by Hervey and Till (13). The electrostatic field is used to control the dispersion and collection of the fibers, and is established by the difference in potential between the electrode and the spray nozzle. The most satisfactory potential difference ranges from 10 to 40 KV, depending on the polymer. The sign of the electrode potential appears to make no difference. Fibers which are produced in an electrostatic field do not "bundle" as they do without the electrostatic charge. In respect to the process described in Part I of this report, the use of an electrostatic field may increase the fineness of the fiber material by dispersing the fibers so that (1) they do not form in bundles which may deposit on the mat, and (2) the recovery of fiber material is increased by reducing the amount of fibers which are drawn together while free-floating and which deposit in streamers or on the chamber floor.

The microfiber webs had both the efficiency and the low pressure drop necessary for very high efficiency air filtration, about equivalent to the "absolute" filter manufactured by the Cambridge Filter Corporation, described later in the report. Efficiency tests of three types of organic fiber filters are given in Table II below. Smoke containing D.O.P. particles  $0.3 \mu$  in diameter at a loading of 50 micrograms per liter of air was passed at a rate of 28 fpm through the filters, and the results were compared with those obtained with "absolute" filter papers. An extremely high initial efficiency was obtained and was attributed to the charged nature of the fibers. The values given in Table II refer to the steady state, and indicate the mechanical efficiency due to the presence of very fine fibers.

Table II

FILTRATION EFFICIENCY OF MICROFIBER WEBS COMPARED WITH  
AEC "ABSOLUTE" FILTER

	Density (g/sq ft)	Thickness (inches)	Pressure Drop (mm H <sub>2</sub> O)	Efficiency (%)
AEC glass and asbestos	21	0.030	115	99.93
AEC all-glass paper	21	0.030	115	99.95
Acrylonitrile/ vinyl chloride copolymer	8.9	0.020	59	97.50
Vinyl chloride/ vinyl acetate copolymer	28	0.070	47	99.81
Acrylonitrile	17	0.040	110	99.80

In about 1957, American Viscose Corporation built a semi-production unit to make plastic microfiber at Marcus Hook, Pennsylvania. They have already issued some licenses under their patents but nonexclusive licenses are still available. It is suggested that organizations investigating the possibility of fabricating material as described in Part I of this report consider the patents held by American Viscose Corporation in order to prevent infringement.

The information was obtained from the patents and monograph cited above, received through Mr. Worth Wade, Director of Patents and Trademarks, American Viscose Corporation.



2. Atomic Laboratories, Inc. (3086 Claremont Avenue, Berkeley 5, California)

Atomic Laboratories, Inc., are producers of "Carbon Wool." This product is composed of carbon in a fibrous form with an extremely high intrinsic fiber density. The ash content is reported to be approximately 0.67%, which is unfortunately too high for the present application. Carbon Wool can be activated to a high level with no appreciable loss in physical properties. The activated fiber has adsorption characteristics equal to those of granular activated carbon, without the high pressure drop which occurs in granular beds.

Cross-link bonded Carbon Wool pads are available with fiber diameters of about 15  $\mu$ . Carbon Wool may also be supplied on order in diameters ranging from 5 to 50  $\mu$ , in rope, pad, cross-bonded filter pads, bulk material of a high density, and in woven forms. It can be prepared on order in any manner such as those employed on vegetable or organic fibers.

The information was obtained from a description issued by Atomic Laboratories, Inc.

3. Cambridge Filter Corporation (738 Erie Boulevard East, Syracuse 3, New York)

Cambridge Filter Corporation manufactures two air cleaning devices: the aerosolve air filter and the "absolute" filter, developed in conjunction with Arthur D. Little, Inc. The Aerosolve filter is used for general industrial purposes. The "absolute" filter was developed for the AEC to remove radioactive particles. The "absolute" filter material is a mixture of glass and asbestos fibers, and is made by a standard wet paper-making process by the Hollingsworth and Vose Company, discussed farther along in the report. It replaces the CWS #6 paper used in their earlier filters. It has a 99.95% collection efficiency for 0.3- $\mu$ -diameter D.O.P. particles. The filter material does not suit the present specifications of Air Force Cambridge Research Center because of purity and ashing requirements. However, the "absolute" filter is recommended for supplying high-purity air to the chamber in which the filters are processed.

The information was obtained through Mr. Paul M. Engle, Manager of Research and Development at the Cambridge Filter Corporation.

4. Cobert S.R.L. Corporation (Ufficio Laboratorio, Milan, Italy)

The Cobert Corporation manufactures filters for automobiles and for air conditioning units. The filter material is manufactured from polyvinyl chloride which is obtained as a byproduct of fabric manufactured by the Montecatini Company, discussed farther along in the report. The fibers are very elastic and rather coarse, having diameters of 0.2 - 0.3 mm. This permits fabrication of compact filters with low air resistance. The filter is covered by Italian and German patents, and a patent was applied for in the U.S. in 1957. The patents include the Cobert contention that particles are precipitated electrostatically. The material is cheap, washable, and acid-base resistant, and retains 95% of particles 1 to 50  $\mu$  in diameter. This retention is sufficient for filtration of air entering automobile engines, and for air conditioning units. At the time that this information was obtained (October, 1958) the Cobert Corporation was testing panels of the material, 50 cm by 50 cm, to be used in industrial air cleaning devices. This company has not considered making experimental filters, but is interested in testing the applicability of its filters for collection of radioactive material. Thinner fibers of polyvinyl chloride could be obtained from the Montecatini Company.

Information was obtained from Mr. Mario Bertone, President of the Cobert S.R.L. Corporation.

5. Connecticut Hard Rubber Company (407 East Street, New Haven 9, Connecticut)

The Connecticut Hard Rubber Company has developed a silicone rubber product called COHRLastic. The product is fibrous in nature, somewhat resembling sponge in properties. The fibers are about 0.5 mm in diameter, and are formed into a mat. The fibers are randomly oriented, resulting in a material which has tensile and tear strength superior to that of silicone sponge and foam. In addition to its improved mechanical

strength, the COMRlastic has high permeability, density in the range of 20 lb/cu ft, good compression-deflection characteristics, and a usable temperature range of -65°F to 500°F. It is presently available from pilot plant equipment in sheets 1/4-in. thick, 9 in. wide, and 6 ft long. It is anticipated that as applications are developed the product will be made in continuous lengths, larger widths, and various thicknesses.

The material is made under exclusive license from American Viscose Corporation, U.S. Patents No. 2,483,404 and 2,483,406. These patents are described in the previous section of the report in which the American Viscose Corporation processes are discussed.

The information was obtained from Mr. Gordon A. Proctor, Sales Representative for the Connecticut Hard Rubber Company.

6. Delbag-Luftfilter, GMBH (Schweidnitzer Strasse, 11-15  
Berlin-Halensee, Germany)

Delbag-Luftfilter is the major manufacturer of industrial air purification equipment in Germany, and has been in operation for over 40 years. American Air Filter used to be the U.S. licensee, but other organizations are being considered at present. Delbag manufactures an industrial filter in which the filter material is folded into a 2 ft by 2 ft frame. The filter material is composed of polystyrene fibers from 0.1 to about 1.0  $\mu$  in diameter, and although the Delbag representatives could not disclose details of their manufacturing process they indicated that it was very similar to the process described in Part I of this report. The polystyrene is dissolved in an organic solvent, and aspirated or forced into an air stream through jets. Samples of Delbag material, and photomicrographs, indicated that the pads, and fibers, were very similar to the unpressed pads developed on this project. The pads are sandwiched between sheets of thin cellulose tissue. The process is patented in Britain, and patents on the manufacturing process and product were applied for in the U.S. about 4 years ago. Delbag has developed a new process which is not covered by the patent applications, and has obtained particularly high filter efficiencies with this new material, using

solid particles and the standard 0.3- $\mu$ -particle-diameter D.O.P. Characteristics of 6 types of polystyrene fiber filter pads made by Delbag-Luftfilter are given in Table III. The tests indicate that Type 4 is the best filter for general use, having a high collection efficiency and a relatively low pressure drop. It is the opinion of the Delbag investigators that filter efficiencies should be tested with a variety of materials. For instance, they found that some filters will take out 99% of D.O.P. aerosol particles (0.3  $\mu$  in diameter), but only 70% of quartz particles.\*

The information on the Delbag-Luftfilter Company was obtained from Dr. Hans Wittemeier, Technical Director, Mr. Fritz Becker, Manager of Research, and Mr. Horst W. Schulze, Export Publicity Director.

7. E. I. Du Pont de Nemours and Co. (Wilmington 98, Delaware)

Du Pont produces woven fabric and felt industrial filters from Nylon, Orlon, Dacron, and Teflon fibers of diameters in the 1- to 10- $\mu$  range. However, these materials have a very high metallic content as a result of intentional additions such as titanium dioxide, and also as a result of contamination during processing. Consequently, it does not appear that these materials could be used directly for high-purity filter manufacture.

A process for preparing felts from synthetic fibers has been described by Lauterbach of the Du Pont Company (14), including felts prepared from polyethylene and Dacron fibers. A modified felting process is used to card, needle, and shrink the material. The author proposes filtration as one of the uses of the felts, with advantages over wool fibers of chemical resistance, heat resistance, and ease of cleaning.

Most of Du Pont's work on high-purity synthetic fibers in the sub-micron diameter range has been done in conjunction with the Naval Research Laboratory, and was discussed previously under Naval Research Laboratory. Du Pont has not found sufficient market potential for these fibers and is not interested in further development work.

\*Tests of efficiency of filters made at Delbag-Luftfilter have been carried out at the Federal Republic Dust Research Institute, at the following address: Staubforschungsinstitut des Hauptverbandes der gewerblichen Berufsgenossenschaften e.V., Langwartweg, Bonn, Federal Republic.

Table III

## CHARACTERISTICS OF POLYSTYRENE FIBER FILTER PADS MADE BY DELBAG-LUFTFILTER

(D.O.P. Aerosol, 0.3- $\mu$ -Diameter Particles)

Flow, cm/sec	Type #1		Type #2		Type #3		Type #4		Type #5		Type #6	
	Resis- tance, mm H <sub>2</sub> O	Pene- tration %	Resis- tance, mm H <sub>2</sub> O	Pene- tration %	Resis- tance, mm H <sub>2</sub> O	Pene- tration %	Resis- tance, mm H <sub>2</sub> O	Pene- tration %	Resis- tance, mm H <sub>2</sub> O	Pene- tration %	Resis- tance, mm H <sub>2</sub> O	Pene- tration %
0.93	--	4	--	3.5	--	0.07	--	0.001	--	--	--	--
1.39	--	10	--	5.5	--	0.2	--	0.001	--	--	--	--
1.85	--	16	--	8	--	0.4	20	0.002	35	30	30	30
2.7	--	14	--	13	10	0.7	28	0.003	56	38	38	38
4.0	--	24	10	16	13	1.1	34	0.007	74	50	50	50
5.3	--	29	12	20	14	1.5	42	0.012	100	62	62	62
7.2	--	42	18	26	22	2.5	58	0.018	138	82	82	82
10.6	4	52	26	30	32	3.2	84	0.032	194	114	114	114
14.2	8	56	35	32	43	4.2	112	0.044	260	152	152	152
17.5	9	57	43	36	52	4.1	137	0.046	316	193	193	193
26.5	13	64	65	38	79	4.5	206	0.052	470	272	272	272
35.3	18	69	86	40	104	4.6	276	0.052		360	360	360
53.0	27	73	130	40	156	4.8	408	0.048		344	344	344
72.0	37	73	176	39	212	4.1	552	0.040				
108	56	71	265	32	318	3.4		0.025				
148	78	69	364	24	435	2.1		0.016				
217	112	62	--	18	--	1.1		0.010				
283	148	49	--	11	--	0.6		0.004				

The information on production of synthetic fibers by Du Pont was obtained from Dr. Walter E. Jordan, Textile Fibers Department, at the Wilmington Experimental station, from Bulletins published by Du Pont, and from the paper cited above.

8. ESB-Reeves Corporation (P.O. Box 8109, Philadelphia 1, Pennsylvania)

The ESB-Reeves Corporation manufactures microporous plastic materials (15). Most of the work to date has been with polyethylene and polyvinyl chloride. The resin is mixed with starch, and the mixture is shaped by any of the techniques used for forming plastics. The mixture is then placed in boiling water, where the starch particles swell and produce a cell structure in the plastic. The starch is then hydrolyzed to sugars with hot, diluted sulfuric acid, and a porous, shaped material remains after the sugars have been leached away. The pore size can be made to vary from 1 to 150  $\mu$ . It is stated that a microporous plastic processed with cornstarch makes an absolute filter for particles with diameters of 1  $\mu$  and greater, and finer particles can be filtered if a finer starch is used.

At the present time this company is doing a considerable amount of development work, some of which is applicable to filtration.

This information was obtained from the article cited above, and from Dr. Howard J. Strauss, Vice-President of ESB-Reeves Corporation.

9. The Goodyear Tire and Rubber Company (Akron 16, Ohio)

The Goodyear Tire and Rubber Company manufactures a shredded polyethylene filter under the trade name "Pliotron." The filter was developed in the course of studies at Goodyear to determine the effectiveness of self-charging electrostatic air filters (16). Goodyear's tests confirmed the choice of polyethylene as the best electrostatic medium for the filter. Shredded polyethylene filters were claimed to have efficiencies of about 99% for smoke particles in the range 0.05 to 0.1  $\mu$  in diameter.

The Pliotron is a permanent filter because it is self-generating, and washing will not affect its electrostatic properties. The polyethylene medium will not shrink or swell, and it is inert to all chemicals found in industrial atmospheres. It will withstand air temperatures up to 212°F. Radioactive materials such as polonium, which emit rays that ionize the atmosphere and produce electromagnetic effects, will discharge electrostatic filters and render them relatively ineffective. A patent was applied for in March, 1950.

The information on Goodyear's "Pliotron" was obtained from publicity releases and the publication cited.

10. Hollingsworth and Vose Company (East Walpole, Massachusetts)

The Hollingsworth and Vose Company manufactures a variety of filter materials, including the Kent cigarette filter. The company's filters are produced by paper-making techniques from mixtures of coarse and fine fibers. They were of the opinion that paper-making techniques would be difficult to apply to organic fibers because of the difficulty of dispersing the fibers in water. They have observed that filter efficiency is higher when the filter contains a distribution of fiber sizes than when the fibers are all of one size. For example, three nozzles were used to obtain the distribution of fiber sizes used by American Viscose Corporation in developing the early "Micronite" filter for Hollingsworth and Vose. They have pilot plant facilities, and in special cases could produce a limited amount of organic filter material by a specified process on a contract basis.

Information on Hollingsworth and Vose Company was obtained from Dr. Harold W. Knudson, Technical Director, and Mr. R. D. Parsons, Engineer. Dr. Knudson and Mr. Parsons worked with the Naval Research Laboratories in developing the hot-melt extrusion process used to produce fibers of thermoplastic materials and with the American Viscose Corporation to produce the "Micronite" cigarette filter. The work of these two organizations is discussed previously in this report.

11. Membranfiltergesellschaft GmbH (Weender Landstrasse 96-102, Göttingen, Germany)

The Membranfiltergesellschaft Company is a subsidiary of Sartorius-Werke, A.G., an old established manufacturer of precision scientific instruments, and is located in the Sartorius plant. "Membranfilters" are made in different porosities from cellulose esters by the same process as Millipore filters in the U.S., discussed below. The characteristics of different types of membranfilters are given in Table IV.

Table IV  
CHARACTERISTICS OF MEMBRANFILTERS

Type	Air Flow, liters/min/100 cm <sup>2</sup> /500mm Water Column	Average Pore Size, $\mu$	Approximate Size of Dust Particles Retainable, $\mu$	Ash Content, mg/sq cm*
AF 600	700-500	5-10	1	0.011
AF 400	500-300	3-5	0.5	0.011
AF 250	300-200	1-3	0.5	0.008
AF 150	200-100	0.7-1	0.3	0.006
AF 100	100-50	0.6-0.8	0.3	0.005
AF 50	50-30	0.5-0.7	0.3	0.005
AF 30	30-15	0.3-0.5	0.1	0.005
AF 15	15-10	0.2-0.3	0.1	0.005
AF medium green	25-20	0.3-0.5	0.1	---

\*Calcium is the primary ash constituent.

High-porosity membranfilters have been used in Germany for quantitative retention of radioactive particles. A filter with an effective area of 150 sq cm is used in these tests, and air is sampled at a rate of 50 cu m/hr. The pressure drop across the membrane is 2000 mm H<sub>2</sub>O. Total radioactivity is determined by standard radiochemical methods. The membrane is 150  $\mu$  thick, and the radioactive material penetrates 10-20  $\mu$ .



Their representatives were of the opinion that the smallest particles retained in these tests are about  $0.1 \mu$  in diameter. They have proved that filters of smaller porosity will retain particles down to  $30 \mu$  in diameter. Tests are being made for Membranfilter at the University of Freiburg by Prof. Sittkus, who is studying quantitative retention of radioactive material on the Type AF 150 filter, and by Prof. Haxel and Dr. Schumann at Heidelberg University. They are studying the retention of radioactive material on the Type AF 400 filter. Information on these studies may be obtained from Prof. Haxel at the following address: Physikalische Institut, Universitat Heidelberg, Philosophenweg 12, Heidelberg, Germany.

Membranfilter has not worked with fiber filters, but they have used materials other than cellulose esters in making their membranes. They have tested polystyrene membranes and, although they could not immediately satisfy the present requirements in respect to pressure drop, they have information on the process (which they could not release) which would allow them to solve the problem very quickly. They could then furnish the material on a contract basis to AFCRC.

Information on the Membranfilter Company was obtained from Dr. K. H. Maier and Miss E. Keese of the Research Department, Mr. Erwin A. Kimbauer, Staff Engineer for the Pall Corporation, assigned temporarily to Membranfilter, and Mr. R. Colmorgen, Export Manager.

#### 12. Millipore Filter Corporation (Bedford, Massachusetts\*)

This company manufactures the well-known Millipore filter, which is produced by precipitation of cellulose esters from solution in organic solvents. The new plant in Bedford was in operation late in 1958. A continuous process has been substituted for the previous batch process in order to improve the reproducibility of the product, and filtered

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\*Present address. Address at time of discussion with Millipore representatives was Watertown, Mass.

air is used to reduce contamination. They claim that there are less than 100 micrograms of residual ash in 47-mm discs of Type HA or AA material. Sheets of Millipore filter material are available in 1 by 9-ft sheets for special research purposes, at \$45 to \$90 a sheet, depending on the type.

Controlled precipitation of polymeric materials is of great interest to Millipore. They have tested their present precipitation process with Nylon and methacrylate, and were (October, 1958) considering the manufacture of a methacrylate filter. They suggested that although heat could be used to strengthen the filter described in Part I of this report, by causing the fibers to fuse together where they cross, the resiliency might be decreased. They suggested that it might be better to dip the pad in a solvent mixture to soften the polystyrene just enough to cause bonding at the contacts, or to strengthen the fiber mat by saturating it with an emulsion of styrene in water, using a dispersing agent.\*

The Corporation was not interested in the production of organic filters from fibers, but they would be very interested in developing suitable organic filters of high purity by their precipitation process.

Information on the Millipore Filter Corporation was obtained from Mr. John H. Bush, President, from Mr. Walter Kenyon, and from their recent Technical Brochure 759.

13. Mine Safety Appliances Company (201 North Braddock Avenue, Pittsburgh 8, Pennsylvania)

During the past 8 years, the technical staff at Mine Safety Appliances Company has evaluated more than 2500 different formulations of fibrous

\*Preliminary tests of these methods of strengthening the Stanford Research Institute fiber filter pad were subsequently made. They were discontinued because of difficulty in controlling the heating and softening of the thin pads containing fine fibers. The use of Dacron mesh was subsequently developed to satisfy the project requirements in respect to strength and ease of handling. More extensive tests of fiber strengthening should be made if filter pads are to be used without reinforcement.

filtering media. Seventeen materials have reached the production stage. All forms of fibrous structures are investigated: papers, cloths, pads, webs, and bats. All methods of construction are considered: felting, weaving, webbing, wet accretion, air laying, flocking, paper making, spinning, and fiber spraying. Many different types of fibers are involved in the investigations, such as mineral, ceramic, glass, metal, animal, plant, carbon, and plastic.

Filter webs are prepared by spraying plastic fibers onto a moving screen with proper controls to regulate fiber diameter, length, distribution, apparent density, and mat thickness. Webs can be made reproducibly with any type of thermoplastic material which is readily liquified in a fast evaporating solvent.

A thin (1/8 in.) web of medium weight (25 g/sq ft) made of Vinyon fibers, approximately 3 to 4  $\mu$  in diameter, prepared by spraying, has a low resistance to air flow in combination with a relatively high dust retention efficiency and holding capacity. At an air velocity of 28 feet/min, there is a pressure drop of only 2 mm of water. The collection efficiency for D.O.P. smoke (particle diameter, 0.3  $\mu$ ) is about 54% at an air velocity of 28 fpm. At this flow rate it has an efficiency of 83% to 99% for dust particles with diameters slightly greater than 0.3  $\mu$ . It is estimated that 1000 cfm of air could be drawn through a 24 by 24 by 6-in. filter made of this material, with an initial pressure drop of 0.1 in. H<sub>2</sub>O (about the same as that of the common furnace filter), that it would hold more than a pound of dust before the pressure drop reached 0.2 in. H<sub>2</sub>O, and that the filter would be 99% efficient when tested by standard methods such as the Bureau of Mines dust test, and the NBS Atmospheric Air Dust Stain Test.

Mine Safety Appliances Company has used the wet accretion method of laying and forming fibers for over 20 years. They consider it to be one of the most practical methods of producing a filter for fluids. The method combines the advantages of the paper-making process, with respect to fiber alignment, with a technique which produces a final filter in one

operation. Gas mask, respirator, and automotive oil filters have been made by this process for a number of years.

Mine Safety Appliances Company is experimenting with wet accreted pads containing a combination of fibers and finely divided activated charcoal, which remove harmful gaseous materials as well as particulate matter from inhaled air. These studies developed out of research over the past 10 years by the U.S. Army Chemical Corps, designed to develop a combination aerosol and gas filter. The latest combat mask developed by the Chemical Corps incorporates a combination fiber and charcoal filter.

A third filter medium developed by MSA is the new Dustfoe Space Filter. It is made of glass microfibers which have been first air-matted and then water-compacted for proper alignment of the fibers. The resulting web is too fragile to handle without reinforcement, and is sandwiched between 2 layers of cotton scrim cloth.

The typical performances of sprayed plastic webs, wet accreted pads, and Dustfoe space filters made by Mine Safety Appliances Company are given in Table V.

The information, including Table V, was obtained from a monograph of Mine Safety Appliances Company by R. B. Evans, Production Line Manager, Ventilation.

14. Ministry of Supply, Physics Section, Chemical Defence Experimental Establishment (Porton Down, Nr. Salisbury, Wilts, England)

At the time of the visit to Porton (October, 1958) the Physics Section there was preparing plastic fiber filters, using a modification of the NRL technique. The production of a polyethylene mat was observed. Polyethylene was heated to 360°C and forced through 7 jets (made from hypodermic needles), using a commercial extruder. Air at 190°C was fed in immediately behind the jets, and the extruded fibers were directed against a movable target. The mats which were produced by this process were very similar in appearance to ours, and the fiber diameters were in

Table V

**FILTER MEDIA PERFORMANCE (TYPICAL)**  
**SUMMARY DATA**

(Mine Safety Appliances Company)

Tests	APPROX. THICKNESS - INCH		Sprayed Plastic Web	Wet Accreted Pad		Dustfree Space Filter Medium
	1/8	3/8				
Pressure drop - mm H <sub>2</sub> O col.						1/32
	at 10.5 fpm		1	40		5
	at 28.0 fpm		2	109		12
	at 60.0 fpm		4	-		25
% D.O.P. penetration - 0.3-μ particle size						
	at 10.5 fpm		26	0.18		12
	at 28.0 fpm		46	0.29		16
	at 60.0 fpm		58	-		30
Weight penetration - milligrams U. S. Bu/Mines Lead Dust Test						
	at 10.5 fpm		0.04 (99.9)*	-		0.03 (99.9)*
	at 28.0 fpm		0.09 (99.8)*	-		0.07 (99.8)*
	at 60.0 fpm		0.17 (99.6)*	-		0.12 (99.7)*
Weight penetration - milligrams U. S. Bu/Mines Silica Dust Test						
	at 10.5 fpm		0.52 (99.6)*	-		0.50 (99.6)*
	at 28.0 fpm		0.96 (99.3)*	-		0.81 (99.4)*
	at 60.0 fpm		1.40 (99.0)*	-		1.10 (99.2)*

Table V (Continued)

Tests	Sprayed Plastic Web	Wet Accreted Pad	Dustfoe Space Filter Medium
% Efficiency NBS Atmospheric Air Dust Stain Test			
at 10.5 fpm	99	-	97
at 28.0 fpm	83	-	78
at 60.0 fpm	76	-	61
at 250.0 fpm	62	-	-
Dust loading characteristics using NBS dust fed at the rate of .5 g/min; 28 fpm air velocity. Total dust load in g/sq ft of filter media required to double the initial resistance	6.38	-	4.25
Useful life against CC 14 in air at concentration of 1000 ppm - in minutes			
at 10.5 fpm	-	120	-
at 28.0 fpm	-	39	-
at 60.0 fpm	-	4	-

% Efficiency

the same range. The same process may be used with polystyrene as with polyethylene. They have developed the following methods for strengthening the filters. The mat may be placed in a mold and heated in an oven for 10 minutes at 100°C. Alternatively, the fiber pads are heated to 80-90°C and rolled with a standard mill-roll. The heating and pressing process causes the fibers to fuse together where they cross, thus greatly increasing the strength of the product. Another variation of the method is to mix fiber products from materials of different melting points. The fibers with the low melting point produce the bonding during the heating treatment. Green and Thomas hold British Patents No. 780,709 and 780,710 on these processes, and have applied for U.S. patents.

The Porton filters are used in making respirators. They have not used the material for high altitude sampling. They have also developed filters from asbestos and wool. The asbestos and wool are carded separately and then blended with two rotating, spiked, concentric drums. The material is then recarded and pressed.

Information at Porton was obtained from Dr. H. L. Green, who is head of the Physics Section and is coauthor of the well known book of Green and Lane, "Particulate Clouds."

15. Montecatini Corporation, "Polymer" Industrie Chimiche S.P.A.  
(Via F. Turati 18, Milan, Italy)

The Polymer organization, which is a member of the Montecatini Corporation, is Italy's largest producer of synthetic fibers.

Their representatives claimed that the Montecatini polyvinyl chloride fiber is the purest PVC obtainable. The fibers are made by an extrusion process, and the finest ones are 1/8 denier or about 1.5  $\mu$  in diameter.\* They claim that cloth made from this material will retain metallic particles, due to the electrostatic charge. They have not attempted to make filters from this material.

\*Denier (of a yarn or fiber) is defined as the weight in g of 9,000 m of the yarn or fiber.

Information was obtained from Mr. G. M. Gallman, Commercial Director of Synthetic Fiber Production at Montecatini.

16. Pittsburgh Coke and Chemical Company (Grant Bldg., Pittsburgh 19, Pennsylvania)

The Pittsburgh Coke and Chemical Company manufacture carbon filters in a variety of forms, including long single filaments, short filaments, felts, and mats of interlaced fibers. The individual fibers are smooth-surfaced, rod-like structures. They are reportedly produced by vapor phase decomposition of hydrocarbons, the fibers being grown on a ceramic surface. The metallic content might be very low if care were taken to eliminate volatile organometallic compounds and subsequent surface contamination. Characteristics of these fibers are given in Table VI, below.

Table VI

CHARACTERISTICS OF PITTSBURGH CARBON FILTERS

Fiber Diameter (Variable, depending on conditions of formation)

Usual diameter: 0.5 - 1.0  $\mu$

Upper range: 4.0  $\mu$

Lower range: 0.1  $\mu$

Fiber length: up to 2 in.

Bulk density: 0.8 lb/cu ft

Surface area: 4 sq m/g

This information was obtained from a bulletin issued by Pittsburgh Coke and Chemical Company on October 1, 1957.

17. Schleicher and Schull (Dassel/Kr. Einbeck, Germany)

The Schleicher and Schull Company manufactures a number of paper products, including filter papers. At the time of discussion (October, 1958) they were manufacturing a limited amount of a filter consisting of



polyvinyl chloride fibers, but this filter will be discontinued by 1960. It is manufactured by a dry process. They are planning the manufacture of a filter from polyvinyl alcohol by a wet process, with organic fiber diameters in the range of 2-3  $\mu$ . Neither of these filters suits the present requirements of particle retention or pressure drop. They also manufacture a glass and asbestos fiber pad by a sintering process, and they have a process for coating cardboard with Membranfilter. This latter product is used mainly as a filter for biologicals. Large particles are retained on the cardboard; small particles on the Membranfilter.

Information was obtained from Mr. Wilhelm Willemer, Chief Engineer for the Schleicher and Schull Company. They are not interested in manufacturing specialized products such as the filter described in Part I of this report, due to limited research and manufacturing facilities.

#### Contributors

The solvent approach to fine-fiber preparation was suggested by C. E. Lapple, Senior Scientist. Efficiency determinations were made by R. C. Robbins and I. S. Yaffe, Physical Chemists. Electron micrographs were prepared and size distribution data were obtained by C. F. Schadt, Physical Chemist. Pilot plant production was carried out by R. Mattos and P. McKeehan, Technicians.



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